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### **Preface**

For centuries, frozen foods have been available to consumers in countries that experience cold winters. In some areas with severe winters such as Alaska, Russia, and others, foods are routinely frozen by leaving them outside. Since 1875, with the development of mechanical ammonia freezing systems, the frozen food industry has grown steadily, especially in the past two decades.

Frozen foods have the advantages of being very close in taste and quality to fresh foods as compared with other preserved or processed foods. Frozen foods are popular and accessible in most developed countries, where refrigerators and freezers are standard home appliances. Nowadays, frozen foods have become essential items in the retail food industries, grocery stores, convenience food stores, fast food chains, food services, and vending machines. This growth is accompanied by the frequent release of new reference books for the frozen food industry.

Several updated books on freezing preservation of foods or frozen foods have been available in the past decade, and most of them are excellent books. The science and technology of food freezing can be viewed from several perspectives:

- Food engineering principles. These principles explain such phenomena as heat and mass transfer, freezing time, convective and conductive processes, and other processes and principles relevant to understanding the dynamics of freezing.
- Food science and technology principles. These principles explain the chemistry and biology of food components, their interactions during processing, and other principles relevant to understanding how foods behave before, during, and after the frozen stage.
- Food manufacturing principles. These principles explain how we can start with a raw ingredient and end with a finished frozen product.
- Food commodities, properties and applications. This approach takes an individual commodity of food (e.g., fruits, vegetables, dairy, muscle foods) and explains the whole spectrum of factors that involve cooling, refrigeration, freezing, and thawing unique to that category of food and its properties. Although the underlying principles are the same, freezing carrots is definitely different from freezing salmon. These data are a combination of the three principles above

and are the basis of our ability to enjoy winter vegetables during summer and 100 flavors of ice cream all year round.

Over the past two decades, books have been published that cover some or all of the topics above. When it comes to books on frozen foods, it is an endless venture. The reason is simple: Every month and every year, food scientists, food technologists, and food engineers witness rapid development in the science and technology of frozen foods. We continually see new knowledge, new equipment, and new commercial applications emerging.

Based on the above premises of principles and applications, the Handbook of Frozen Foods uses the following approaches to covering the data:

- *Principles*. Chapters 1 through 8 cover principles applicable to the processing of frozen foods, such as science, technology, and engineering. Topics include the physical processes of freezing and frozen storage, texture, color, sensory attributes, and packaging.
- *Meat and poultry*. Seven chapters (Chapters 9–15) discuss freezing beef and poultry meat, covering operations, processing, equipment, packaging, and safety.
- Seafoods' Chapters 16 through 21 discuss frozen seafoods, covering principles, finfish, shellfish, secondary products, HACCP (Hazards Analysis and Critical Control Points), and product descriptions.
- *Vegetables*. Five chapters (Chapters 22–26) discuss frozen vegetables, covering product descriptions, quality, tomatoes, French fries, and U.S. grades and standards.
- Fruits. Chapters 27 through 29 discuss frozen fruits and fruit products, covering product descriptions, tropical fruits, and citrus fruits.
- Special product categories. Chapters 30, 31, and 32 provide details on some popular products: frozen desserts, frozen dough, and microwavable frozen foods.
- Safety. Chapters 33 through 36 discuss the safety of processing frozen foods covering basic considerations, sanitation of a frozen food plant, risk analysis in processing frozen desserts, and U.S. enforcement tools for frozen foods.

This volume is the result of the combined effort of more than 50 contributors from 10 countries with expertise in various aspects of frozen foods, led by an international editorial team. The book contains eight parts and 36 chapters organized into eight parts. In sum, the approach for this book is unique and makes it an essential reference on frozen food for professionals in government, industry, and academia.

We thank all the contributors for sharing their experience in their fields of expertise. They are the people who made this book possible. We hope you enjoy and benefit from the fruits of their labor.

We know how hard it is to develop the content of a book. However, we believe that the production of a professional book of this nature is even more difficult. We thank the production team at Marcel Dekker, Inc., and express our appreciation to Ms. Theresa Stockton, coordinator of the entire project.

You are the best judge of the quality of this book.

Y. H. Hui Paul Cornillon Isabel Guerrero Legarreta Miang H. Lim K. D. Murrell Wai-Kit Nip

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## **Contributors**

Alma D. Alarcon-Rojo Universidad Autónoma de Chihuahua, Chihuahua, Mexico

José Felipe Alemán-Escobedo Centro de Investigación en Alimentación y Desarrollo, A. C., Hermosillo, Sonora, Mexico

S. M. Alzamora Universidad de Buenos Aires, Buenos Aires, Argentina

María Cristina Añón Universidad Nacional de La Plata, La Plata, Argentina

**Sheryl A. Barringer** Department of Food Science and Technology, The Ohio State University, Columbus, Ohio, U.S.A.

**D. Bermúdez** Universidad de las Américas—Puebla, Puebla, Mexico

Geneviève Blond ENSBANA-Université de Bourgogne, Dijon, France

Phil J. Bremer Department of Food Science, University of Otago, Dunedin, New Zealand

**Juan Pedro Camou-Arriola** Centro de Investigación en Alimentación y Desarrollo, A.C., Hermosillo, Sonora, Mexico

Salvador Castillo Miguel Hernandez University, Orihuela, Spain

**Roberto S. Chamul** California State University, Los Angeles, Los Angeles, California, U.S.A.

Harvey T. Chan, Jr. HI Food Technology, Hilo, Hawaii, U.S.A.

Philip Cheng-Ming Chang National Taiwan Ocean University, Keelung, Taiwan

Hsing-Chen Chen National Taiwan Ocean University, Keelung, Taiwan

Chau Jen Chow National Kaohsiung Institute of Marine Technology, Kaohsiung, Taiwan

**Patti C. Coggins** Department of Food Science and Technology, Mississippi State University, Mississippi State, Mississippi, U.S.A.

J. Fernández-López Miguel Hernandez University, Orihuela, Spain

H. Douglas Goff Department of Food Science, University of Guelph, Guelph, Ontario, Canada

Natalia F. González-Méndez Centro de Investigación en Alimentación y Desarrollo, A.C., Hermosillo, Sonora, Mexico

**Richard W. Hartel** Department of Food Science, University of Wisconsin–Madison, Madison, Wisconsin, U.S.A.

Y. H. Hui Science Technology System, West Sacramento, California, U.S.A.

B. Jamilah University Putra Malaysia, Selangor, Malaysia

Shann-Tzong Jiang National Taiwan Ocean University, Keelung, Taiwan

William L. Kerr Department of Food Science and Technology, University of Georgia, Athens, Georgia, U.S.A.

Christopher C. Lai Pacteco Inc., Kalamazoo, Michigan, U.S.A.

Alain Le Bail ENITIAA-UMR GEPEA, Nantes, France

**Tung-Ching Lee** Department of Food Science, Rutgers University, New Brunswick, New Jersey, U.S.A.

Martine Le Meste ENSBANA-Université de Bourgogne, Dijon, France

Alberto Edel Leon Universidad Nacional de Córdoba, Córdoba, Argentina

Jens Liesebach Department of Food Science, University of Otago, Dunedin, New Zealand

Miang H. Lim Department of Food Science, University of Otago, Dunedin, New Zealand

Ana Guadalupe Luque-Alcaráz Centro de Investigación en Alimentación y Desarrollo, A.C., Hermosillo, Sonora, Mexico

**Domingo Martínez-Romero** Miguel Hernandez University, Orihuela, Spain

J. Mateo-Oyagüe Universidad de León, León, Spain

Janet E. McFetridge Department of Food Science, University of Otago, Dunedin, New Zealand

H. Mújica-Paz Universidad Autónoma de Chihuahua, Chihuahua, Mexico

**Athapol Noomhorm** Asian Institute of Technology, Pathumthani, Thailand

**Bonnie Sun Pan** National Taiwan Ocean University, Keelung, Taiwan

J. A. Pérez-Alvarez Miguel Hernandez University, Orihuela, Spain

M. L. Pérez-Chabela Universidad Autónoma Metropolitana, Mexico City, Mexico

Edith Ponce-Alquicira Universidad Autónoma Metropolitana, Mexico City, Mexico

**Stephen C. Ridley** College of Agriculture, Food, and Environmental Science, University of Wisconsin–River Falls, River Falls, Wisconsin, U.S.A.

M. R. Rosmini Universidad Nacional del Litoral, Santa Fe, Argentina

Peggy Stanfield Dietetic Resources, Twin Falls, Idaho, U.S.A.

Alfonso Totosaus Universidad Autónoma del Estado de Hidalgo, Hidalgo, Mexico

A. Valdez-Fragoso Universidad Autónoma de Chihuahua, Chihuahua, Mexico

Daniel Valero Miguel Hernandez University, Orihuela, Spain

**Punchira Vongsawasdi** King Mongkut's University of Technology Thonburi, Bangkok, Thailand

J. Welti-Chanes Universidad de las Américas—Puebla, Puebla, Mexico

**Louise Wicker** Department of Food Science and Technology, University of Georgia, Athens, Georgia, U.S.A.

Kit L. Yam Rutgers University, New Brunswick, New Jersey, U.S.A.

**Libertad Zamorano-García** Centro de Investigación en Alimentación y Desarrollo, A. C., Hermosillo, Sonora, Mexico

Hua Zhao Rutgers University, New Brunswick, New Jersey, U.S.A.

## 1

## **Freezing Processes: Physical Aspects**

Alain Le Bail ENITIAA-UMR GEPEA, Nantes, France

#### I. INTRODUCTION

This chapter presents the freezing process. Selected models permitting estimates of the freezing time are proposed and discussed. These models are based on the classical model established by Plank; improvements of this model are presented and discussed. The different types of freezing processes used in the industry are then presented. Blast freezing is probably the most popular freezing process, but other concepts such as contact freezing are also used in a wide range of applications. The thermal contact resistance existing between the refrigerated surface and the product is often neglected; a focus is proposed on this aspect. The discussion ends with an evaluation of the freezing rate.

#### II. FREEZING PROCESS

#### A. Heat Transfer During Freezing

The heat transfer phenomenon involved in freezing of biological material is basically nonlinear heat transfer. The latent heat of water represents a large amount of heat that has to be removed from the foodstuff. Generally, a high freezing rate is desired in order to obtain numerous small ice crystals. Nevertheless, this is not always the case. For example, consider frozen dough, for which a slower freezing gives a better preservation of yeast activity. Freezers can be classified in two families; batch freezers, for which a given amount of product will be frozen in the same batch, and continuous freezers, which can be operated in a production line. The refrigeration system used allows classifying freezers in two other subfamilies: freezers using cryogenic fluids such as carbon dioxide or liquid nitrogen, and freezers using a mechanical refrigeration unit and a secondary refrigeration fluid (air, brine, etc.). Mechanical refrigeration units are used for a large majority of industrial freezers. Cryogenic fluid will be used for special applications requiring (a) minimal investment, (b) specific use (i.e., meat grinding), or (c) high freezing rate. Heat transfer conditions and thus the freezing rate are closely related to the type of freezer.

#### B. Freezing Time

A basic analytical model has been proposed by Plank (1941) assuming that (a) the initial temperature of the product is equal to the phase change temperature, (b) phase change occurs at constant temperature, and (c) all thermophysical properties and heat transfer coefficients are constants. Consequently, the initial cooling and final cooling after freezing are not taken into account. The freezing time given by the Plank formula is proposed in Eq. (1).

$$t = \frac{\Delta H \cdot \rho \cdot X}{(T_{a} - T_{f}) \cdot N} \cdot \left[ \frac{1}{h} + \frac{X}{4\lambda_{F}} \right]$$
(s)

where t is freezing (thawing) time (s);  $\Delta H$  is enthalpy difference over the freezing plateau  $(\mathbf{J} \cdot \mathbf{kg}^{-1})$ ;  $\rho$  is density of the frozen food  $(\mathbf{kg} \cdot \mathbf{m}^{-3})$ ;  $T_a$  is medium temperature (K or °C);  $T_f$  is initial freezing temperature (K or °C);  $T_f$  is thermal conductivity of the food in the frozen state  $(\mathbf{W} \cdot \mathbf{m}^{-1} \cdot \mathbf{K}^{-1})$ ;  $T_f$  is the transfer coefficient  $T_f$  is characteristic dimension (m); and  $T_f$  is coefficient (see Table 1).

Based on this first approach, several authors attempted to improve the accuracy of the freezing (or thawing) time calculation. Ramaswamy et al. (1984) proposed a review of these equations. Nagoaka et al. (1955) proposed Eq. (2), which takes into account the amount of heat to be removed during the pre- and postfreezing periods. In this equation,  $\Delta H$  represents the enthalpy difference between the initial temperature ( $T_i$ ) of the product and the final temperature at the end of freezing ( $J \cdot kg^{-1}$ ).

$$t = \left[1 + 0.008^* T_i\right] \frac{\Delta H \cdot \rho}{(T_f - T_a)} \cdot \left[\frac{PX}{h} + \frac{RX^2}{\lambda_F}\right]$$
(s) (2)

with P and R geometric factors as defined in the table below.

Geometry	X	P	R
Slab	Thickness	0.5	0.125
Cylinder	Diameter	0.25	0.0625
Sphere	Diameter	0.167	0.0416

Levy (1958) proposed an expression extrapolated from the model of Nagoaka that differs mainly in that the temperature difference between initial and final conditions is explicitly taken into account, the enthalpy difference being considered between the initial temperature ( $T_i$ ) of the product and the final temperature at the end of freezing ( $J \cdot kg^{-1}$ )

 Table 1
 Coefficient of the Plank Formula

Geometry	L	N
Slab	Thickness	2
Cylinder	Diameter	4
Sphere	Diameter	6

(Eq. 3). The International Institute of Refrigeration (1972) proposed Eq. (3), which is once again very similar to the model of Nagoaka. This time it is the enthalpy difference that is taken into account between the initial freezing temperature and the final temperature  $T_c$  ( $\Delta H$  = enthalpy between  $T_f$  and final temperature of the product  $T_c$  in  $J \cdot kg^{-1}$ ) (Eq. 4).

$$t = \left[1 + 0.008(T_{\rm i} - T_{\rm f})\right] \frac{\Delta H \cdot \rho_F}{(T_{\rm f} - T_{\rm a})} \cdot \left[\frac{PX}{h} + \frac{RX^2}{\lambda_{\rm F}}\right]$$
(s)

$$t = \frac{\Delta H \cdot \rho_{\rm F}}{\left(T_{\rm ifp} - T_{\rm a}\right)} \cdot \left[\frac{PX}{h} + \frac{RX^2}{\lambda_{\rm F}}\right] (s) \quad t = \frac{\Delta H' \cdot \rho'}{\left(T_{\rm f} - T_{\rm a}\right)} \cdot \left[\frac{Pd}{h} + \frac{Rd^2}{\lambda_{\rm F}}\right] (s) \tag{4}$$

Cleland and Earle (Cleland et al., 1979) used an approach based on numerical models and experimental results. The Plank equation is proposed in nondimensional form using the Fourier number *Fo* based on the *P* and *R* factors and on the Biot number (*Bi*) and Stefan number (*Ste*).

$$F_0 = \frac{P}{BiSte} + \frac{R}{Ste} \tag{5}$$

$$Ste = \frac{\Delta H \text{ between } T_{\text{f}} \text{ and } T_{\text{a}}}{\Delta H \text{ between } T_{\text{f}} \text{ and } T_{\text{final}}}$$
 (6)

$$Bi = \frac{hX}{2\lambda_{\rm F}} \tag{7}$$

These authors introduced a dimensionless Plank number [Pk, Eq. (8)] and defined new coefficients  $P^*$  and  $R^*$  of the new Plank equation for a slab.

$$Pk = \frac{\Delta H \text{ between } T_i \text{ and } T_f}{\Delta H \text{ between } T_f \text{ and } T_{\text{final}}}$$
(8)

$$P^* = 0.5072 + 0.2018Pk + Ste(0.3224Pk + \frac{0.0105}{Bi} + 0.0681)$$
(9)

$$R^* = 0.1684 + Ste(0.2740Pk + 0.0135) \tag{10}$$

$$t = \frac{\Delta H \text{ (between } T_{\text{f}} \text{ and } T_{\text{final}}) \cdot \rho}{(T_{\text{f}} - T_{\text{a}})} \cdot \left[ \frac{R^* X}{h} + \frac{R^* X^2}{\lambda_{\text{F}}} \right] \text{(s)}$$
(11)

This equation was acceptable for initial temperature  $T_i < 40^{\circ}\text{C}$  and medium temperature between  $-15^{\circ}\text{C} < Ta < 40^{\circ}\text{C}$ , heat transfer coefficients between  $10 < h < 500 \,\text{Wm}^{-2} \cdot \text{k}^{-1}$  and maximum slab thickness of 12 cm. For an infinite cylinder, they found Eqs. (12) and (13).

$$P^* = 0.3751 + 0.0999Pk + Ste(0.4008Pk + \frac{0.0710}{Bi} - 0.5865)$$
 (12)

$$R^* = 0.0133 + Ste(0.0415Pk + 0.3957)$$
(13)

And for a sphere,

$$P^* = 0.1084 + 0.0924Pk + Ste(0.2310Pk - \frac{0.3114}{Bi} + 0.6739)$$
 (14)

$$R^* = 0.0784 + Ste(0.0386Pk - 0.1694) \tag{15}$$

These latter were applicable for 0.155 < Ste < 0.345, 0.5 < Bi < 4.5, and 0 < Pk < 0.55. The numerical parameters were obtained from experiments realized with thylose gels (77% w.c.). The accuracy of these relationships for freezing time prediction were within  $\pm 3\%$  for slabs,  $\pm 5.2\%$  for infinite cylinders and  $\pm 3.8\%$  for spheres Ramaswamy and Tung (1984) established a new model extrapolated from the previous one. They use a regression approach:

$$t = [0.3022C(T_{\rm i} - T_{\rm f}) + L + 2.428C'(T_{\rm f} - T_{\rm c})] \frac{\rho'}{(T_{\rm f} - T_{\rm a})} \cdot \left[ \frac{Pd}{h} + \frac{Rd^2}{\lambda_{\rm F}} \right] (s)$$
 (16)

With C and C' = specific heat of the foodstuff respectively before and after freezing  $(J \cdot kg^{-1} \cdot K^{-1})$  and L = enthalpy over the freezing plateau  $(J \cdot kg^{-1})$ . This formula was established in the following conditions:

1°C < 
$$T_i$$
 < 25°C, −18°C <  $T_c$  < −10°C, −178°C <  $T_a$  < −18°C,  
13.9 <  $h$  < 68.4 W · m<sup>2</sup> · k<sup>-1</sup>.

Other expressions are available in the literature but the one proposed above can be considered as a good basis. The accuracy can be greatly improved by using numerical models for which extensive studies have been done (Cleland, 1990). These models allow us to take into account the time-dependent heat transfer coefficient and the temperature. Modern software is now available to realize this type of modeling without major difficulties.

## III. CONVECTIVE PROCESSES: AIR FREEZING, BRINE FREEZING, CRYOGENIC FREEZING

In the case of convective freezing, air, a cryogenic fluid (mainly liquid nitrogen), or a brine can be used as refrigerant. In the case of air, it can be admitted that the air velocity is in the range of 1 to  $5 \,\mathrm{m} \cdot \mathrm{s}^{-1}$  for most industrial application, leading to the effective heat transfer coefficient in the range of 10 to  $50 \,\mathrm{W} \cdot \mathrm{m}^{-2} \mathrm{k}^{-1}$  between the medium and the product. Large-scale spiral freezers have been developed by equipment companies and are widely used in the industry. Individual quick freezing (IQF) consists of freezing small products individually with a high air speed (i.e.,  $1-5 \,\mathrm{m}\cdot\mathrm{s}^{-1}$ ). Freezing of larger products can be realized with blast air but will yield a low freezing rate and thus a low quality in terms of ice crystal size; plate freezers are preferred. Some specificity in terms of air flow pattern have been developed in order to reduce water loss by dehydration (i.e., counter flow or partial counter flow with air inlet a mid position between entrance and exit). Higher air velocity can also be imposed in a local section such as the entrance of the freezer. This has been developed for small products (few centimeters thick). It improves heat transfer and permits a superficial freezing which will minimize mass losses by reducing partial vapor pressure at the surface of the product. This partial superficial freezing is also called crust freezing or cryomechanical freezing (Macchi, 1995; Agnelli et al., 2001); it can be realized

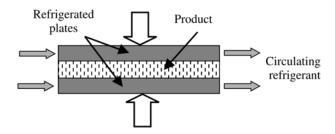
by using a liquid nitrogen bath in which the products are floating for a short period before traveling either into a conventional belt freezer at follow or in the vicinity of the cryogenic bath to gain the benefit of the vaporized gas for the final freezing (see Mermelstein, 1997). Freezing in a brine will yield a much higher heat transfer coefficient than in blast air. The product can be wrapped; in this case, a brine made of CaCl<sub>2</sub>, NaCl, propylene glycol, ethanol or mixtures of them can be used (Venger et al., 1990). In the case of unwrapped products (Lucas et al., 1999) the freezing process can be combined with a soaking effect. Soaking resulted in a salt concentration at the surface of the product and prevented freezing in an external layer (a nonfrozen layer of ca. 1 mm has been observed [Lucas et al., 1999b]). Brine freezing is also used for small products such as shrimp to prevent excessive water loss by dehydration and eventually to enhance solute intake; in the case of shrimp, for example, a brine solute is generally a mixture of salt and sugar. One major drawback of the immersion technique is that the brine concentration is changing during the process, requiring a specific adjustment of it during the treatment. Undesirable side effects may occur such as spoilage of the product by the brine (requiring filtering and cleaning of the brine) and cross-contamination of pathogenic microorganisms, as shown by Berry et al. (1998).

The use of solid carbon dioyde as a refrigerant is an intermediate solution between contact freezing (contact of the solid flakes of  $CO_2$ ) and convective freezing (convective heat transfer between the sublimated  $CO_2$  and the product). An optimal design will result in a temperature of the gaseous  $CO_2$  as high as possible.

#### IV. CONDUCTIVE PROCESSES: CONTACT FREEZERS

Contact freezing can be considered as a mass production process that has a relatively high freezing rate. IQF and cryogenic refrigeration will yield yet higher freezing rates. It is widely used in the industry to produce slabs of frozen foods such as fish filets and mashed vegetables. As for any freezing process, the geometry of the product will rule the freezing rate as described by the Plank equation (Plank, 1941). Two classes of contact freezers can be defined, continuous and batch systems. In batch systems, the product is usually frozen from both sides, by plane heat exchangers applying a certain pressure against the product (Fig. 1). Continuous systems are usually operated by applying a thin product on a refrigerated surface and by scraping it off after freezing. Two concepts have been developed, namely rotating drum freezers and linear belt freezers (Marizy et al., 1998).

In batch plate freezers, the product is usually installed in a cardboard box containing a plastic film or pouch. It can eventually be installed directly against the refrigerated surface, but this will create a problem in removing the frozen product at the end of the process. Rotating drum freezers (Fig. 2) have been developed in the industry to freeze



**Figure 1** Contact freezer: batch system.

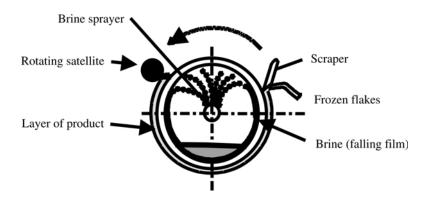
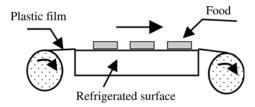


Figure 2 Contact freezer: rotating drum freezer for liquid and viscous food.

liquid or viscous products or even solid products such as fish filets. In this case, the product is directly applied against a rotating metallic drum refrigerated from the inside by a brine, for example. Such a process has been modeled by Marizy et al. (1998). The product is applied on one side (thickness between 1 mm and a few cm) and is scraped after a rotation. Madsen (1983) studied drum freezing of codfish and showed that this process improved the storage stability of cod relative to those frozen in a plate freezer. Recent literature on drum freezer technology mainly concerns patents. Some of these patents are related to heat transfer improvement between the refrigerant and the drum, such as Reynolds (1993) in the case of boiling refrigerant (R22 type). Specific patents are related to food preparation and processing such as a patent (Hoogstad, 1988) for preparing tea or coffee extracts destined to freeze drying, a patent (Dalmau, 1987) for citrus fruit freezing, a patent (Roth, 1982) for freezing and forming meat patties, and a patent (W.a.A., 1969) for shrimp processing. Several refrigeration techniques are used: boiling refrigerant (i.e., Reynolds (1993)), cryogenic refrigerant (Anonymous, 1980) or brine. Cryogenic refrigerant such as liquid nitrogen is an expansive solution, as gas is emitted to the ambience. Nevertheless, its very low phase change temperature permits it to achieve high heat flux and thus fast freezing. Wentworth et al. (1968) presented a development for increasing the efficiency of the cryogenic fluid distribution thanks to a jacket. Anonymous (1980) describes a process in which the disadvantage of the stagnant cryogenic fluid at the lower part of the drum is used as an advantage to remove the product from the drum (owing to the thermal shock caused by the sudden cooling).

Machinery using the linear design (Fig. 3) has been recently developed and proposed on the market, while the rotating design has been used several years to freeze liquid or semiliquids foods. The linear continuous contact freezer consists of a refrigerated surface on which a plastic film is sliding, or on which food is frozen on a mobile refrigerated surface. In the former, the product is applied onto the film and is frozen during its translation on the refrigerated surface. After freezing, the product is removed from the film, which is discarded. Additional refrigerating effect is usually added by allowing



**Figure 3** Contact freezer: linear system with a sliding plastic film.

refrigerated air on top of the product. This kind of process is not extremely efficient owing to thermal contact resistance between the product and the refrigerated surface. The plastic film represents a first thermal resistance. Moreover, the uncontrollable deformation of the product will result in the apparition of an air film between the cold surface and the plastic film. Nevertheless, this process remains very handy in achieving a superficial freezing of the product preventing dehydration during conventional freezing.

Single-side contact freezing is of course less efficient than double-side contact freezing. Nevertheless, a continuous process is highly desirable in the industry in order to minimize contamination by handling and also for productivity. Maltini (1984) studied the application of solid foods to contact freezing processes and did a comparison with air freezing. He suggested that the food should be regular in shape to ensure contact of greater than  $20 \, \mathrm{mm}^2 \cdot \mathrm{g}^{-1}$ . Donati (1983) did a similar study and compared the drum freezing technique with several other freezing processes.

#### V. SCRAPED FREEZERS

The scraped heat exchanger has been adapted to the case of freezing ice cream. It typically consists of a rotating drum equipped with one or two blades. The rotating drum is installed in a refrigerated vessel. The blades allow the scraping of the ice crystals formed onto the inner surface of the refrigerated drum. They also permits whipping of the air inside the mix. Indeed, a certain overrun (amount of air entrapped in the final ice cream) must be obtained to ensure an acceptable texture of the ice cream. For this purpose, a certain back pressure must be applied at the exit of the system (between ca. 100 and 500 kPa). Fat and air structures in ice creams have been investigated by several authors (Bolliger et al., 2000). The temperature of the refrigerated surface, the formulation, the back pressure, and the rotating speed will interact with the degree of fat destabilization and foam structure.

Straight blades are usually used. Helical blades aiming to propel the ice cream mix toward the exit of the system has been evaluated by Myerly (1998). More recently, a new design of continuous freezer has been developed by Windhab et al. (1998). This system has been developed from a twin screw extruder that has been adapted for the freezing of ice creams. The enhanced local shear stresses acting in the extrusion channel resulted in improved microstructure in comparison with conventional scraped heat exchanger. This process, known as cold extrusion, can yield an ice cream at a much lower temperature than a conventional scraped heat exchanger. Thus the ice cream obtained with such a freezer cannot be used to fill forms or mold but does not need any further conventional hardening.

#### VI. THERMAL PERFORMANCE OF FREEZING PROCESSES

The thermal performance of a given freezing process is related to the overall energy consumption required to cool down a given product from an initial temperature down to a final one. An accurate evaluation of this parameter is difficult because it has to take into account the type of refrigerating system (mechanical compression, cryogenic) being considered, the geometry of the product, the freezing rate, the final temperature, and the balance that will be considered between the refrigeration in the freezing process per se and the refrigeration load that will be held by the storage system. A first approach can be realized by comparing the heat transfer coefficient between the refrigerated medium

(convective freezing) and the surface (contact freezing). The order of magnitude of the effective heat transfer coefficient as defined by Eq. (17) for convective process or by Eq. (18) for contact freezing are summarized in Table 2.

$$U = h = \frac{\Phi}{(T_{\rm F} - T_{\rm S})}$$
 with  $T_{\rm S}$  and  $T_{\rm F} = T$  @ surface and fluid (17)

$$U = h = \frac{\Phi}{(T_{\rm F} - T_{\rm S})}$$
 with  $T_{\rm S}$  and  $T_{\rm F} = T$  @ surface and fluid (17)
$$U = \frac{1}{\rm TCR} = \frac{\Phi}{(T_{\rm RS} - T_{\rm FS})}$$
 with TCR = thermal contact resistance (18)

 $T_{\rm RS}$  and  $T_{\rm FS}=T$  @ refrigerated and food surface.

One can see from Table 2 that assuming a "perfect" thermal contact in the case of contact freezing is not acceptable. If the product is applied directly onto the refrigerated surface, heat transfer coefficients as high as 500 to 1000 can be expected, but are a function of process parameters as detailed by LeBail et al. (1998). A drum freezer used to freeze mashed vegetables (broccoli in the present case) was studied. A parameter study showed that the thermal contact resistance was higher for lower surface temperature. This supposes that the mechanical stress in the product during freezing (stretching owing to ice formation) interacts with the quality of the mechanical and therefore the thermal contact between the surface and the frozen product. The roughness of the metallic surface is also an important parameter. Specific study of LeBail (unpublished data) showed that a factor of 10 can be observed between a smooth and a rough surface of stainless steel (ca.  $70 \,\mathrm{W}\cdot\mathrm{m}^{-2}\cdot\mathrm{K}^{-1}$  and  $700 \,\mathrm{W}\cdot\mathrm{m}^{-2}\cdot\mathrm{K}^{-1}$  for the rough and the smooth surface, respectively). The presence of packaging drastically deteriorates the heat transfer. A plastic film seems to reduce slightly the heat transfer coefficient, whereas the presence of cardboard results in a heat transfer coefficient that can be as low as  $20 \,\mathrm{W} \cdot \mathrm{m}^{-2} \cdot \mathrm{K}^{-1}$ (Creed et al., 1985), which is comparable to blast air freezing.

The thermal efficiency of the freezing process is thus highly related to the geometry of the product, its physical state (solid, liquid), and the process that is considered. A high

**Table 2** Effective Heat Transfer Coefficients U for Convective and Conductive Freezing Processes as Defined by Eqs. (17) and (18)

Process	Conditions	$\mathrm{U}\;(\mathrm{W}\cdot\mathrm{m}^{-2}\cdot\mathrm{K}^{-1})$	Ref.
Convective	Blast air	10–50	
Convective	Brine	50-500	
Convective	Liquid nitrogen, smooth cylinders, warming regime $U = \frac{1860}{(T_F - T_S)} + 125 \left[ \mathbf{W} \cdot \mathbf{m}^{-2} \cdot \mathbf{K}^{-1} \right]$	120–200	Macchi, 1995
Convective	Liquid nitrogen, strawberry, meat balls (experimental)	170–230	Agnelli and Mascheroni, 2001
Conductive	Drum freezer with direct contact, sample = mashed broccoli mean value ranging between (vs. process parameters)	214 1000–166	Marizy et al., 1998; LeBail et al., 1998
Conductive	Plate freezer: sample: copper block; 310 kPa pressure		Creed et al., 1985
	No wrapping	481	
	1 layer polyethylene film	278	
	Corrugated fiberboard + polyethylene	20	

freezing rate is usually desired except for some specific products (i.e., bread dough). The packaging plays a major role. It permits a reduction of water loss but has a negative effect on the heat transfer rate. At present, individual quick freezing of meat products, for example, chicken breast, is much used in the industry. Recent studies have pointed to immersion freezing in brine, even though some problems will necessarily occur for the treatment of the brine. Contact freezing is also widely used and is well adapted for mass production. The presence of packaging is required for obvious handling reasons (removal of the frozen product from the refrigerated surface) but has a very negative impact on the efficiency of the process. Direct freezing in cardboard should be avoided.

#### VII. FREEZING RATE

The freezing rate is central to the final quality of frozen foods. A slow rate results in cell dehydration and large ice crystals that might damage the texture of a food. A fast freezing rate prevents the migration of water into the extracellular spacing and yields fine and numerous ice crystals. Side effects such as the increasing of the concentration of the remaining aqueous solution might affect the integrity of cell membranes or of proteins. The freezing rate is a very general statement used most of the time to compare freezing conditions. The freezing rate is numerically presented in two ways in the literature: Plank (1941) proposed an expression of the freezing rate evaluated as the velocity of the phase change front (dimension/time). The International Institute of Refrigeration (IIR, 1972) defined the nominal freezing time as the duration between 0°C and 10°C above the initial freezing temperature. Based on this definition, several researchers calculated the freezing rate by a ratio of temperature difference and the respective duration (Eq. (19) in K/time). This approach, which can be called temperature formulation, yields a freezing rate unit in  $K \cdot s^{-1}$  or in  $K \cdot min^{-1}$  (practical unit). The approach proposed by Plank (1941) will be called the Plank formulation and yields freezing rate in  $m \cdot s^{-1}$  or  $cm \cdot h^{-1}$  (practical unit). Plank calculated the velocity of the phase change front by deriving the expression of the freezing time. This yielded Eq. (19a-c), respectively, for slab, cylinder, and sphere with x = distance from center (slab), r = radius, and  $r_0 =$  outer radius of the geometry.

$$w(x) = \frac{(T_{\rm a} - T_{\rm f})}{\rho \cdot \Delta H \cdot \left[\frac{1}{h} + \frac{x}{\lambda_{\rm F}}\right]} \qquad (m \cdot s^{-1})$$
(19a)

$$w(r) = \frac{(T_{\rm a} - T_{\rm f})}{\rho \cdot \Delta H \cdot \left[\frac{r}{r_o \cdot h} + \frac{r}{\lambda_{\rm F}} \operatorname{Ln}\left(\frac{r_o}{r}\right)\right]} \qquad (m \cdot s^{-1})$$
(19b)

$$w(r) = \frac{(T_{\rm a} - T_{\rm f})}{\rho \cdot \Delta H \cdot \left[\frac{r^2}{r_{\rm o}^2 \cdot h} + \frac{r(r^2/r_{\rm o} - r)}{\lambda_{\rm F}}\right]} \qquad (\text{m} \cdot \text{s}^{-1})$$
(19c)

$$F_{\rm T}(r) = \frac{T_1 - T_2}{t_1 - t_2} \qquad (\mathbf{K} \cdot \min^{-1})$$
 (19d)

In the case of the temperature formulation, a beginning criterion and an ending criterion for freezing must be defined [subscripts 1 and 2 in Eq. (19)]. LeBail et al. (1996, 1998b) showed that the freezing rate value is dependent on these criteria. Thus the use of the freezing rate from Plank expression or the evaluation of the freezing rate from the ratio of the lower thickness by the corresponding freezing time [i.e., determined by the nominal freezing time (IIR, 1972)]. In this latter case, a mean freezing rate will be obtained.

A high freezing rate might result in cracks in the product. Shi et al. (1999) reported that stress as high as 2 MPa (20 atm) can be reached during the freezing of biological tissue. This result was obtained from a mathematical model (viscoelastic model coupled to a thermal model) developed to study the freezing of a sample of potato (17.8 mm diameter). A frozen mantle first appears at the surface of the product. Meanwhile, the formation of ice at the core will yield an increase of the pressure. Radial and circumferential stresses develop during freezing. Rapid temperature drop (i.e., cryogenic freezing with surface temperature down to  $-196^{\circ}$ C for liquid nitrogen) will induce higher stress, which can be as high as 1.5 MPa, whereas freezing in a medium at  $-40^{\circ}$ C yields stress in the range of 1 to 0.5 MPa (Shi et al., 1999). Even though the temperature of the frozen external mantle is far from the glass transition, the tensile failure strength, which was around 0.5 MPa for potato, might be passed, leading to cracks. On the other hand, a depression of the initial freezing point due to a pressure increase will result in a partial thawing (Otero et al., 2000) leading to a release of the stress.

#### VIII. CONCLUSION

This chapter offers a general presentation of the freezing processes including evaluation of the freezing time. A focus proposed on the surface heat transfer coefficient includes contact freezing. It shows that an infinite heat transfer coefficient can't be assumed in this process, which is widely used in the industry.

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## 2

## **Principles of Freeze-Concentration and Freeze-Drying**

#### J. Welti-Chanes and D. Bermúdez

Universidad de las Américas-Puebla, Puebla, Mexico

#### A. Valdez-Fragoso and H. Mújica-Paz

Universidad Autónoma de Chihuahua, Chihuahua, Mexico

#### S. M. Alzamora

Universidad de Buenos Aires, Buenos Aires, Argentina

In freeze-concentration and freeze-drying processes, water is first frozen in the material. Ice is removed by mechanical means during freeze-concentration, leaving a concentrated liquid, while ice is removed by sublimation in freeze-drying, yielding a dried material. The removal of water by these methods yields high-quality products, but in both processes it is a very expensive operation owing to the high consumption of energy. Knowledge of the theoretical principles behind these processes is necessary for minimization of detrimental changes, operating strategies, and optimization purposes. Thus the fundamental aspects of freeze-concentration and freeze-drying are presented in this chapter.

#### I. FREEZE-CONCENTRATION

#### A. Introduction

Freeze-concentration is the term used to describe the solute redistribution in an aqueous solution with an initial relatively low concentration by the partial freezing of water and subsequent separation of the resulting ice [1,2]. Freeze-concentration is based on the freezing temperature-concentration diagram (Fig. 1) [3].

It is necessary briefly to review the physicochemical changes that occur during a freezing process before relating them to the freezing of foods. The phase diagram (Fig. 1) allows identifying different phase boundaries in a mixture. It consists of the freezing curve (AB), solubility curve (CE), eutectic point (E), glass transition curve (DFG), and conditions of maximal freeze-concentration. The freezing curve corresponds to solution—ice crystals equilibrium. Along this curve, as water is removed as ice, the concentration of solute increases during the freeze-concentration process. The solubility curve represents equilibrium between the solution and supersaturated solution in a rubbery state. The freezing and solubility curves intersect at the eutectic point E (Ce, Te), which is defined as

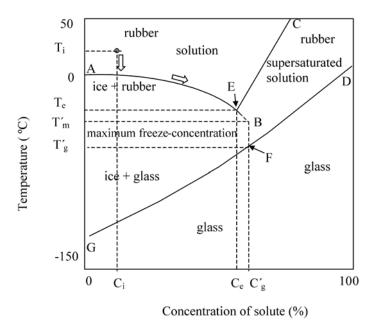
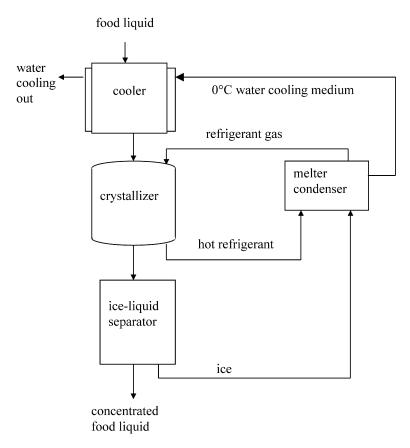


Figure 1 Typical solid-liquid state diagram for a food system.

the lowest temperature at which a saturated solution (liquid phase) can exist in equilibrium with ice crystals (solid phase). The water content at point E is the unfreezeable water. Below  $T_e$  only ice crystals embedded in a solute—water glass exist. The point F ( $C_g', T_g'$ ) lower than point B ( $C_g', T_m'$ ) represents a characteristic transition in the state diagram. The glass transition curve (DFG) represents the glass—rubber transition of the solute—water mixture, and the type and concentration of the solute and the temperature define it. Above the DFG curve, solutions are in an unstable rubbery or liquid state; below the DFG curve, solutions transform into the glassy state (amorphous solid). The maximum freeze-concentration (maximum ice formation) only occurs in the region above  $T_g'$ , but below the equilibrium ice melting temperature of ice ( $T_m'$ ) [4,5]. The liquid solute—water mixture is the maximum freeze-concentrated and has become glassy. The glass transition temperature of this unfrozen glassy mixture is designated  $T_g'$ , and  $C_g'$  is the solid content of this glass [3–7]. Figure 1 also shows the aqueous solution with initial concentration and temperature  $C_i$  and  $T_i$  undergoing freeze-concentration.

#### B. Freeze-Concentration System

A typical freeze-concentration system (Fig. 2) consists of three fundamental components: (a) a crystallizer or freezer, (b) an ice-liquid separator, a melter-condenser, and (c) a refrigeration unit. In the freeze-concentration system, the solution is usually first chilled to a prefreezing temperature in a cooler (Fig. 2), and then the solution enters the crystallizer where part of the water crystallizes. Cooling causes ice crystal growth and an increase in solute concentration. The resulting mixture of ice crystals and concentrated solution is pumped through a separator where crystals are separated and the concentrated solution is drained off. Ice crystals are removed and melted by hot refrigerant gas. The final products are cold water and concentrated solution, which flow separately [1,8].



**Figure 2** Schematic diagram for the freeze-concentration process of foods.

#### 1. Crystallizers

The heat of crystallization can be taken out directly or indirectly. In direct-contact crystallizers, the original solution is allowed to get in contact with the refrigerant, and heat is withdrawn by vacuum evaporation of part of the water, usually at pressures below 3 mm Hg, and by evaporation of the refrigerant. The refrigerants (CO<sub>2</sub>, C<sub>1</sub>–C<sub>3</sub> hydrocarbons) form icelike gas hydrates, which sequester water at temperatures above 0°C. A disadvantage of this method is that part of the aromas will be lost during the evaporation. Direct heat removal is applied in seawater desalinization but is not suitable for liquid foods, owing to the aroma losses and deterioration of the product by the refrigerant. In crystallizers with indirect heat removal, the refrigerant (R22 or ammonia) is separated from diluted solution by a metal wall. So crystallization takes place on chilled surfaces, from which ice crystals are removed by a scraper. This kind of process has been used commercially for orange juice and coffee concentration [1,9].

#### 2. Separators of Ice-Concentrated Solution

The separation of ice crystals from concentrated solutions can be performed by the use of presses, centrifuges, and wash columns, operating in either batch or continuous mode.

Hydraulic and screw presses are used for pressing ice-concentrated slurries to form an ice cake. Pressures around  $100\,\mathrm{kg/cm^2}$  are needed to avoid occlusion of solids in the cake, which is the limiting factor of this method. Since the presses are completely closed, aroma loss is negligible [1,9].

Ice and concentrated solutions may be separated by centrifugation at about 1000 G. Centrifugation must be conducted under inert atmospheres to reduce oxidation and aroma loss. Solute losses may occur if concentrated solution remains adhered to the crystal surface, but washing of the cake with water will minimize such losses. This washing stage renders the centrifugation operation more efficient than pressing [1,2,9].

In washing columns, the ice-solution mixture is introduced at the bottom of the tower, and the solution is drained off. The crystals move toward the top of the column in countercurrent to the wash liquid, which is obtained by melting part (5–3%) of the washed crystals leaving the column. In this process the loss of dissolved solids with the ice is less than 0.01%, and aroma losses are negligible. Wash columns are preferred in freeze-concentration of low-viscosity liquids such as beer and wine [1,8,9].

#### C. Influence of Process Parameters

Crystallization is the main step in freeze-concentration, so it is very important to obtain large and symmetrical crystals. Large crystals can be more easily separated from the concentrated solution. Large crystals also reduce the loss of solutes due to occlusion and adherence to the small crystals [1,8]. During crystallization, two kinetic processes take place: the formation of nuclei and the growth of crystals. Nucleation is the association of molecules (at some degree of subcooling) into a small particle that serves as a site for crystal growth. Once a nucleus is formed, crystal growth is simply the enlargement of that nucleus. Nucleation and growth of crystals are dependent on solute concentration, bulk supercooling, residence time of the crystals in the crystallizer, freezing rate, molecular diffusion coefficient of water, and heat transfer conditions. These factors should be carefully controlled to regulate crystal formation [2,10].

#### 1. Solute Concentration

In general, an increase in solute concentration produces an increase in nucleation and a decrease in the growth velocity of the ice crystals and in the mean diameter of the crystal. At critical concentration, solutes may solidify along with ice and are difficult to separate. Practical maximum concentrations for freeze-concentration are between 45–55% range [1,9,10].

#### 2. Bulk Supercooling

Supercooling is the driving force responsible for the creation of crystal nuclei and their growth. The nucleation rate is proportional to the square of the bulk supercooling. At high bulk supercooling values, the nucleation rate decreases, owing to the inhibition of molecular mobility. Crystal growth exhibits a first-order dependence of the bulk supercooling [1,9,10].

#### 3. Residence Time of the Crystals in the Crystallizer

At constant bulk supercooling and solute concentration, the crystal size is proportional to the crystal residence time. At short residence times the crystals produced are very small [1,10].

#### 4. Freezing Rate

A high freezing rate results in a strong local supercooling near the heat-removing interface, thus leading to high nucleation rates and to small crystals. A decrease in freezing rate results in large, uniform crystals with small surface area [1,10].

#### 5. Molecular Diffusion Coefficient of Water

A decrease in the value of the molecular diffusion coefficient of water results in a decrease in diameter of the crystals [1].

#### 6. Heat Transfer Conditions

The growth rate of ice crystals increases greatly as the rate of heat removal is increased, until some very low sample temperature is reached, at which mass transfer difficulties (as high viscosity) cause the growth rate to decline. Very large uniform crystals require large exchange surface at relatively high temperatures [1,2,9].

#### 7. Viscosity of the Liquid

Viscosity increases markedly as concentration increases, ice crystals grow very slowly at high viscosity, and large crystals become difficult to separate. The maximum concentration obtainable in freeze-concentration depends on the liquid viscosity. Generally, concentration can be carried out to the point where the slurry becomes too viscous to be pumped. For essentially all liquids, this viscosity limit is encountered before eutectic point formation occurs (Fig. 1). The viscosity of cold concentrated liquid and ice is very high, and agitation, which is necessary for proper crystal growth, becomes more difficult [9,10].

In all the ice separators, capacity is inversely proportional to the viscosity of the concentrate and directly proportional to the square of the mean diameter of the crystals as expressed by the equation

$$Q = \frac{\Delta Pgd_{\rm e}^2}{0.2\,\mu l} * \frac{\varepsilon^3}{(1-\varepsilon)^2} \tag{1}$$

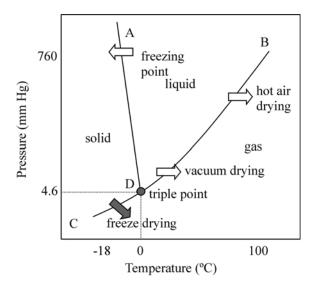
where Q is the draining rate from the crystal bed (cm³/cm²s);  $\Delta P$  is the pressure difference exerted over the bed by compression or by centrifugal or pressure drop of the filtrate (kg/cm²);  $d_e$  is the diameter of the crystals (cm);  $\mu$  is the viscosity of the liquid (poise); l is the thickness of the bed (cm); g is the gravity acceleration (cm/s²); and  $\varepsilon$  is the volume fraction in the bed filled by the liquid phase [1].

#### II. FREEZE-DRYING

#### A. Introduction

Freeze-drying, or lyophilization, is the process of removing water from a product by sublimation and desorption [11]. Sublimation is the transformation of ice directly into a gas without passing through a liquid phase. Sublimation occurs when the vapor pressure and the temperature of the ice surface are below those of the triple point (4.58 mm Hg, 0°C), as shown in the pressure–temperature phase diagram of pure water (Fig. 3) [12].

The phase diagram of Fig. 3 is separated by lines into three regions, which represent the solid, liquid, and gaseous states of water in a closed system. The points along the separating lines represent the combinations of temperature and pressure at which two states are in equilibrium: liquid–gas equilibrium (DB line), liquid–solid equilibrium (DA



**Figure 3** Pressure–temperature phase diagram of pure water.

line), and solid—gas equilibrium (DC line), which is of main concern in freeze-drying. Point D represents the only combination of temperature and pressure at which all three states of water are simultaneously in equilibrium, and it is called the triple point [4,12].

Freeze-drying can also be conducted at moderated pressures and even at atmospheric pressure. The principle of this process is to produce a vapor pressure difference as large as possible by blowing dry air over the frozen material. In practice, the process is very long because of the low mass and energy transfer rates, but problems related to the application of vacuum do not exist, resulting in an important reduction of operation costs [13,14].

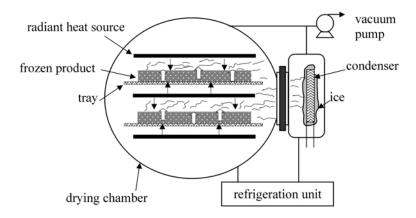
Freeze-drying is used to obtain dry products of higher quality than those obtained with conventional drying methods. Freeze-dry products have high structural rigidity, high rehydration capacity, and low density, and they retain the initial raw material properties such as appearance, shape, taste, and flavor. This process is generally used for the dehydration of products of high added value and sensitivity to heat treatments, produced by the pharmaceutical, biotechnological, and food industries.

Compared to air drying processes, which remove water in a single stage, freezedrying is an expensive process, since it takes large operation times and consumes large amounts of energy. Energy is required to freeze the product, heat the frozen product to sublimate ice, condense water vapor, and maintain the vacuum pressure in the system [15,16].

#### B. Basic Components of a Freeze-Dryer

The typical freeze dryer consists of a drying chamber, a condenser, a vacuum pump, and a heat source (Fig. 4).

The drying chamber, in which the sample is placed and heating/cooling take place, must be vacuum tight and with temperature-controlled shelves. The condenser must have sufficient condensing surface and cooling capacity to collect water vapor released by the product. As vapors contact the condensing surface, they give up their heat energy and turn into ice crystals that will be removed from the system. A condenser temperature of  $-65^{\circ}$ C



**Figure 4** Simple schematic representation of a freeze-dryer system.

is typical for most commercial freeze-dryers. The vacuum pump removes noncondensable gases to achieve high vacuum levels (below 4 mm Hg) in the chamber and condenser. The heating source provides the latent heat of sublimation, and its temperature may vary from -30 to  $150^{\circ}$ C [13,17,18].

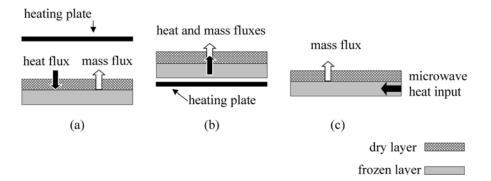
#### C. Freeze-Drying Stages

Freeze-drying involves three essential stages: initial freezing, primary drying, and secondary drying. The objective of the freezing stage is to freeze the mobile water of the product. The product must be cooled to a temperature below its eutectic point, which is the temperature and composition combination that produces the lowest point at which a product will freeze. Freezing has an important influence on the shape, size, and distribution of the ice crystals and thus on the final structure of the freeze-dried product. In the primary drying, the frozen product is heated under vacuum conditions to remove frozen water by sublimation, while the frozen product is held below the eutectic temperature. During the primary drying, approximately 90% of the total water in the product, mainly all the free water and some of the bound water, is removed by sublimation [19,20]. In the secondary drying, bound water (unfrozen) is removed by desorption from the dried layer of the product, achieving a product that should contain less than 1–3% residual water. This final stage is performed by increasing the temperature and by reducing the partial pressure of water vapor in the dryer [12,20].

The secondary drying stage requires 30 to 50% of the time needed for primary drying because of the lower pressure of the remaining bound water than free water at the same temperature, yielding a slow process. Freeze-drying is complete when all the free and bound water has been removed, resulting in a residual moisture level that assures desired structural integrity and stability of the product [12,16].

#### D. Heat and Mass Transfer in Freeze-Drying

During the freeze-drying operation, a coupled heat and mass transfer process occurs within the product: energy is transported to the sublimation zone and water vapor is generated. In contrast with mass transfer, which always flows through the dry layer, heat transfer can take place by conduction through the dry layer (Fig. 5a) or through the frozen



**Figure 5** Basic types of freeze-drying.

layer (Fig. 5b), and by heat generation within the frozen layer by microwaves (Fig. 5c) [12,20]. Microwaves are used as a heat source for drying because they are able to penetrate deeply into the product, giving a more effective and uniform heating [21,22].

Figure 6 illustrates a frozen food sample in the form of a slab, with a frozen and a dried porous layer, undergoing one-dimensional freeze-drying [12,23]. The interface between the dried and the frozen layers is referred to as the sublimation or ice front, and it is assumed to move at a uniform rate. The vapor flows through the pores and channels.

In case heat is supplied through the dry layer, the heat flux to the ice front is given by

$$q = k_{\rm d} \frac{(T_{\rm e} - T_{\rm f})}{(1 - x)L} \tag{2}$$

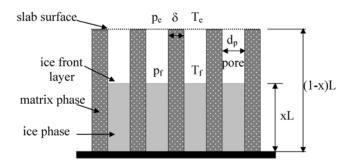
where q is the heat flux (J/m<sup>2</sup> s),  $k_d$  is the thermal conductivity of the dry layer (W/m K),  $T_e$  is the temperature at slab surface (°C),  $T_f$  is the temperature of sublimation front (°C), L is the thickness of the slab (m), and x is the relative height of the ice front.

If heat is transferred through the frozen layer,

$$q = k_{\rm f} \frac{\left(T_{\rm p} - T_{\rm f}\right)}{xL} \tag{3}$$

where  $k_f$  is the thermal conductivity of the frozen layer (W/m K) and  $T_p$  is the heating plate temperature (°C).

If vapor flows in the pores mainly by Knudsen diffusion, the collisions with the dry walls are numerous compared with collisions between water molecules. Thus the rate of ice



**Figure 6** Schematic representation of freeze-drying of a slab.

sublimation  $(N_{\rm W}, \, \text{kg/s m}^2)$  is given by

$$N_{\rm W} = \varepsilon D_{\rm K} \frac{M_{\rm w}}{RT} \frac{p_{\rm f} - p_{\rm e}}{(1 - x)L} \tag{4}$$

where  $\varepsilon$  is the volume fraction of ice,  $D_{\rm K}$  is the Knudsen diffusion coefficient of water vapor (m<sup>2</sup>/s),  $M_{\rm w}$  is the molecular mass of water (kg/mol), R is the gas constant (J/mol K), T is the absolute temperature (K),  $p_{\rm f}$  is the vapor pressure at the ice front, and  $p_{\rm e}$  is the vapor pressure at the slab surface (Pa).

The Knudsen diffusion coefficient is related to pore diameter  $(d_p)$  and temperature by

$$D_{\rm k} = \frac{2}{3} d_{\rm p} \left(\frac{2RT}{\pi M_{\rm w}}\right)^{0.5} \tag{5}$$

At the ice front,  $T_f$  represents the sublimation temperature, and by assuming it to be in equilibrium, it is related to vapor pressure  $(p_w)$  by the Clausius-Clapeyron equation,

$$\ln(p_{\rm w}) = 28.9 - \frac{6138}{T_{\rm f}} \tag{6}$$

From a mass balance for water vapor around the drying slab,

$$N_{\rm W} = \varepsilon \rho_{\rm ice} \frac{d(xL)}{dt} \tag{7}$$

where  $\rho_{ice}$  is the density of ice (kg/m<sup>3</sup>).

Assuming that all supplied heat is used for sublimation of ice, the enthalpy balance gives

$$q = \Delta H_{\rm s} N_{\rm W} \tag{8}$$

where  $\Delta H_s$  is the latent heat of sublimation (J/kg).

For temperature differences not too large, the Clausius-Clapeyron equation can be linearized, and the above equations can be solved analytically. The following expressions can be derived for the total drying time:

$$t = \frac{\alpha L^2}{D_K} (1 + \beta D_K) \tag{9}$$

in which

$$\alpha = \frac{1}{2} \rho_{ice} \frac{RT_{\rm f}}{M_{\rm w}} \frac{1}{(p_{\rm f} - p_{\rm e})} \tag{10}$$

If heat transfer takes place through the dry layer, then  $\beta = 0$ ; and if heat transfer occurs through the frozen layer,

$$\beta = \varepsilon \frac{M_{\rm w}^2 \Delta H_{\rm s}^2 p_{\rm f}}{R^2 T_{\rm f}^3 k_{\rm ice}} \tag{11}$$

where  $k_{\text{ice}}$  is the thermal conductivity of ice (W/m K).

Several mathematical equations describing mass and energy transfer have been developed for modeling the freeze-drying process. Such models account for the removal of frozen water only (sublimation model) or for the removal of frozen and bound water

(sorption sublimation model). These models also examine the methods of supplying heat and the diffusion mechanisms, describe steady or non-steady state processes, or analyze both transfers under various processing conditions. Some models have been found to describe accurately experimental drying rates and freezing times. However, a major problem in the application of some models is the requirement of reliable data on thermal and mass transport properties of food materials such as diffusivity within the porous medium, Knudsen diffusion, water vapor concentration in the dry layer, porosity, effective thermal conductivity, permeability, etc. [15,19,21,24].

#### E. Influence of Parameters

A number of operation variables influence the performance of the freeze-drying process and the characteristics of the final product.

#### 1. Freezing

The freezing rate has an important influence on the ice configuration and thus on the final structure of the freeze-dried product. Slow freezing rates allow the growth of large ice crystals, leading to larger pores, to higher mass flow, and thus to shorter freeze-drying times [12,23].

#### 2. Heat Flux

Heat flux that reaches the product is an important factor to reduce the drying rate. However, if the drying proceeds too rapidly (high heat flux), the product may melt, collapse, or be blown out of the container [18]. This may cause degradation of the product and will change the physical characteristics of the dried material. Excessive heat may cause the dry cake to char or shrink. The heating rate can be optimized during operation modifying conveniently product temperatures in the dried zone and at the sublimation front [15,25].

#### 3. Chamber Pressure

The most important operation variable in the freeze-drying process is chamber pressure. The pressure both controls the mean of the sublimation temperature and modifies the transport parameters that influence the kinetics of vapor removing. At a given temperature, a decrease of the pressure in the drying chamber reduces the vapor pressure at the product's external surface  $(p_e)$ , thus the driving force  $(p_f - p_e)$  for drying is enlarged, and the total drying time is reduced. Nevertheless, at low pressures, the sublimation rate may be limited by the transport of water vapor through the product, if the transport of water vapor falls in the free molecular flow regime [21,25,26].

Chamber pressure affects the transport properties, thermal conductivity, and water vapor diffusivity. Thermal conductivity of the dry layer is higher at higher chamber pressures, within the range of freeze-drying operation, resulting in high heat transfer rates from the surface to the ice front. Water vapor diffusivity through the dry layer is, however, less at higher chamber pressures, producing low mass transfer rates. So, when pressure is low (low sublimation temperature), freeze-drying is often a heat-controlled process, but at relatively high pressures freeze-drying becomes a mass-controlled process. In most situations, the drying rate is limited by the rate of heat transfer through the dry layer [25–27].

#### 4. Temperature

Aroma diffusivities are very similar to that of water when the water content is still high; therefore maintaining low temperatures during primary drying will reduce aroma losses. The melting point of products has a significant effect on the selection of operation pressures, since this is a fundamental factor for the sublimation temperature. Normally, a vacuum must be kept so high that no melting occurs in the product during the process, and a true freeze-drying or sublimation takes place. If the temperature of ice in the condenser is higher than product's temperature, water vapor will tend to move toward the product, and drying will stop [26,28].

When freeze-drying temperature is high enough, the product cake suffers a drastic loss of its structure and is said to have undergone collapse. Collapse affects aroma retention, caking and stickiness, rehydration capacity, and final moisture of the product. A collapse temperature  $(T_c)$  is related to the glass transition temperature  $(T_g)$ , which in turn depends on temperature and moisture content (Fig. 1). At temperatures higher than  $T_g$ , the viscosity of the amorphous matrix decreases drastically, this decrease being a function of  $(T - T_g)$ . As the viscosity decreases to a level that facilitates deformation, the matrix can flow, and structural collapse can occur. A critical viscosity in the range of  $10^5-10^8$  Pas has been reported to observe collapse [20,29].

#### III. CONCLUSIONS AND RECOMMENDATIONS

Despite the reduced use at the industrial level of freeze-concentration and freeze-drying processes within the food area, both are important to obtain high quality products. Deep knowledge of the fundamentals of phase changes of water in foods and of the effect of the variables on the processes' effectiveness and cost can open new opportunities for the application of both processes to obtain high-quality preserved foods.

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# **3** Principles of Frozen Storage

#### Geneviève Blond and Martine Le Meste

ENSBANA-Université de Bourgogne, Dijon, France

The use of freezing for food preservation has rapidly developed; the fact that products, mainly meat and fish, could be stored for considerable periods and served, after thawing, as fresh products are at the origin of its development as one of most common methods of food preservation.

Freezing implies two linked processes: a lowering of temperature and a change of phase of water from liquid to solid. Both processes tend to reduce rates of physical and chemical changes and might be expected to enhance the shelf life of the products.

At the same time, quality degradation during storage remains the most common problem for manufacturers and for bringing food freezing to the fullest possible extent. So it is vital to understand well the essentials of the technology. Freezing seldom improves the quality of food products; the raw material quality is of primordial importance, and this quality must be preserved during processing and storage. There is no single universal rule governing frozen food preservation; just as with optimal freezing rates, which vary from product to product, the storage time depends not only on the temperature but also on the type of product and packaging. Most physical and chemical reactions are slowed with the decrease in temperature, but they are not stopped at common storage temperatures. The product deterioration during cold storage is a slow, continuous, cumulative, and irreversible process.

The physical properties of food products change dramatically depending on water availability and temperature. The major assumption relating to quality, and thus shelf life, is that stability is maintained in the glassy state. In a glass, the diffusion of solutes and degradation reactions should be strongly limited, and long-time stability during storage expected. The shelf life of frozen food products should be largely controlled by the physical state of the freeze-concentrated fraction produced by the ice separation. It is desirable to know better how this physical state changes with temperature.

#### I. PHYSICAL STATE AS A FUNCTION OF TEMPERATURE

#### A. Physical Changes During the Freezing Process

According to the freezing process presented in the first chapter, it is obvious that the freezing of food is more complex than the freezing of water. The removal of water by separation of ice produces supersaturation of dispersed substances. For that to occur,

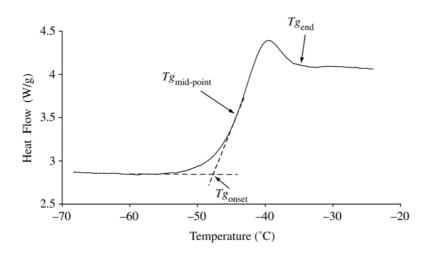
temperature of the product must be lowered sufficiently. The chemical potential of water is reduced by increasing concentration in solutes. The ice formation requires a decrease in temperature, which depends on the product composition.

For practical purposes, the freezing process is considered complete when most of water at the center of the food product has been converted into ice. At  $-15^{\circ}$ C, more than 80% of total water is transformed into ice [1]. The system is segregated into a crystalline phase of pure water and an amorphous domain, which contains solutes and residual water. As the temperature decreases, the viscosity of the interstitial fluid increases rapidly as a result of both concentration increase and temperature decrease. When the viscosity reaches  $10^{11}$ – $10^{12}$  Pa·s, a solidification (vitrification) occurs, and the concentrated phase surrounding the ice crystals becomes a glass. The temperature at which this transition appears is called Tg', the glass transition temperature of the maximally freeze-concentrated system [2]. The freezing of water is stopped at this temperature; the water still unfrozen at Tg' is often called "unfreezable" water.

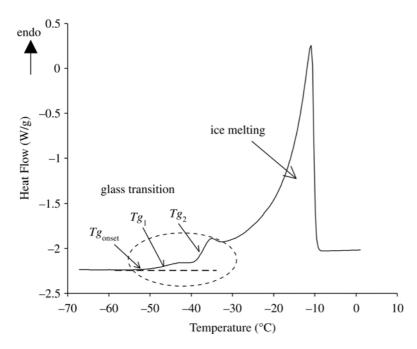
Since the water content of foods is often close to 80–90%, its behavior during the freezing process can be considered as that of an aqueous solution. The proposed model is often a sucrose solution, which could be representative of the freezing behavior of a wide range of solutions when no solute crystallizes during cooling. If cooling is slow enough compared to the kinetics of ice formation, the concentration in the liquid phase depends only on the temperature for a given product and is independent of its initial water content. As a consequence, Tg' is also independent of the initial water content. Fruits, fruit juices, vegetables like tomatoes, and ice creams present very similar thermal changes. The equilibrium temperatures and concentrations vary with the nature and composition of the product.

#### B. Determination of *Tg'*

Differential scanning calorimetry (DSC) is the technique most often used to measure the glass transition temperature; this transition appears as a typical heat capacity jump on the DSC traces and can be characterized by different temperatures (Fig. 1). In the presence of ice, the glass transition, which is visible just before the ice-melting peak on the heating scans, appears more complex, presenting a larger heat capacity change than predicted



**Figure 1** DSC thermogram of a 80% sucrose solution (cooling and heating rates: 10°C/min).



**Figure 2** DSC thermogram of a frozen sucrose solution (50% w/w) (cooling and heating rates:  $10^{\circ}$ C/min).

from the solid content in macromolecular solutions [3]. For frozen solutions of small solutes (sugars, polyols), the thermograms show a two-step endotherm (Fig. 2). These features appear to be very similar to those observed with synthetic polymers and are interpreted as a glass transition with associated enthalpy relaxation [4,5]. There is still no agreement on how the DSC data should be interpreted. The reported Tg' values, according to some authors, is the temperature of the midpoint or the onset of the first transition; for others, it is the midpoint of the second step. They are called, respectively,  $Tg_{\text{onset}}$ ,  $Tg_1$ , and  $Tg_2$  on Fig. 2. This explains the variations in published Tg' values [6].

Because of the ambiguity of the Tg' determination from simple DSC traces, it was suggested to use simplified state diagrams of solute-water blends, which represent their different states in the low-temperature range. The two curves, corresponding, respectively, to the equilibrium liquids or ice melting curve (Tm curve), and the kinetically controlled glass transition curve (Tg curve), are drawn. The diagram representing the various states of the sucrose–water system as a function of concentration and temperature is appropriate to illustrate the demonstration (Fig. 3). The curve Tm represents the temperature at which ice begins to separate as a function of initial concentration, or the concentration of the freeze-concentrated phase as a function of temperature. For all points on this curve, the freeze-concentrated phase is in equilibrium with ice; its partial water vapor pressure is equal to that of ice at the same temperature. For soluble solutes, the temperature of the glass transition depends on the water content; it decreases when the water content increases. The intersection of the two curves could provide a better estimation of Tg' [7]. The freezing of water is stopped at this temperature; the concentration of the maximally freeze-concentrated phase is called Cg' and its water content considered as the "unfreezable" water.

There are obvious problems with the accuracy of the Tm and Tg curves to build a precise diagram. The experimental Tm values as obtained by DSC for the most

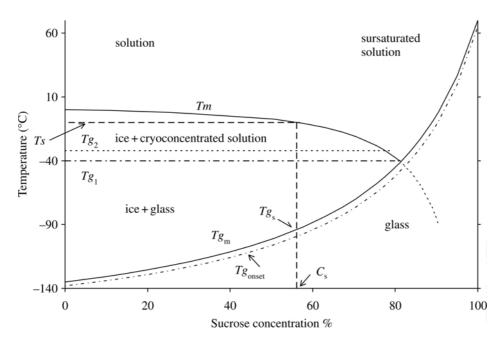


Figure 3 State diagram of sucrose-water binary. (Adapted from Ref. 8.)

concentrated solutions are considered as more or less reliable; an extrapolation of the Tm curve can be subjective; therefore the curve represented in Fig. 3 is derived from excess properties and solid-liquid equilibrium using a UNIQUAC model [8]. For the Tg curve, the experimental data taken into account can be the temperature of the beginning  $Tg_{\rm onset}$ , of the middle of the transition  $Tg_{\rm mid-point}$  (value most often used) or even of the end of the transition. (See Fig. 1 for sucrose solution.) The cooling/heating rates contribute to some changes in temperature due to the time dependence of the glass transition feature. The DSC values of  $Tg_{\rm onset}$  and  $Tg_{\rm 1}$  of frozen sucrose solutions (in Fig. 2) were found to be close to the intersection of the Tm curve with the  $Tg_{\rm onset}$  and  $Tg_{\rm mp}$  curves in Fig. 3. The Tg'-Cg' coordinates vary from  $-45^{\circ}$ C, 82.2% to  $-41^{\circ}$ C, 81.2% [8] depending on the Tg value taken into account. Using the method of optimal annealing temperature, Abblet and coauthors have obtained very similar data  $-40^{\circ}$ C, 82% for Tg'-Cg' [9].

The liquid–glass conversion must be considered to occur in a temperature range that is characteristic of the fraction forming the glassy matrix. The coordinates Tg'-Cg' can be far away from the eutectic point predicted by equilibrium thermodynamics, for example, with sucrose–water binary Te, Ce-14°C, 64%.

Thermomechanical spectroscopy (DMTA) studies of frozen sucrose solutions show that the change in mechanical properties becomes perceptible at  $Tg_{\rm onset}$  as seen by DSC and that a maximum in the loss modulus (E'' or G'') occurs at a temperature between  $Tg_1$  and  $Tg_2$  [10]. Apart from its scientific interest, the sucrose–water phase diagram (Fig. 3) could be considered as representative for a wide range of products containing low molecular weight solutes; very similar diagrams are reported for some fruits [11].

# C. Storage Conditions and Shelf Life of Frozen Foods

The choice of the storage temperature is of considerable practical importance in the frozen food industries. It is the major variable, which can affect stability; a product retaining a good quality for months at  $-20^{\circ}$ C can lose it in a few days at  $-10^{\circ}$ C. Moreover, it is well

demonstrated that frozen foods stored at fluctuating temperatures have not the same shelf life as products stored at constant temperatures [12,13]. The weakest links of the frozen food chain are handling time, temporary storage, and transport temperatures. The last link of the chain, the retail shops, is often critical. Frozen products delivered to the retail outlets are not always placed in refrigeration immediately; the problem includes temperature increases during defrosting. In Europe, where food stores are not open 24 h per day as in the United States, using night covers and programming the defrosting during the night may help to minimize the temperature fluctuations.

In order to ensure product quality, temperature control is necessary throughout the cold chain, and the required temperature must be maintained from production to consumption. European and international regulations concerning the storage temperature of frozen foods set  $-18^{\circ}$ C as the highest temperature during storage and distribution, although many studies have shown that slow degradation of the product quality will occur even at this temperature. This temperature must be stable and maintained at all points in the product (therefore at the surface); possible brief upward fluctuations of 3°C maximum during transport are admitted, as well as a tolerance of 3°C during local distribution and in retail display cabinets [14]. To maintain an optimal quality, the products are generally held at temperatures colder than this in primary cold stores (-20,  $-25^{\circ}$ C).

There is a clear effect of temperature on storage life, with lower temperatures resulting in extended shelf life; but  $-18^{\circ}$ C corresponds to an optimum between the financial costs and the shelf life of frozen foods. As it is the same temperature for all frozen products, the marketing shelf life varies from 6 months to 2 years (Table 1) [1].

At this temperature frozen foods are not fully frozen, nor inert, the Tg' of most products being below  $-18^{\circ}$ C. Table 2 shows the Tg' of some food products or more exactly the temperatures corresponding either to the second step of the transition (called  $Tg_2$  on Fig. 2) or to the midpoint for a single transition. The Tg data for solutions of numerous simple ingredients (sugars, polysaccharides, proteins) can be found in an article by Levine and Slade [23]. It must be emphasized that these temperatures, and consequently the glassy state, will be readily attained by a very limited number of frozen food products at temperatures commonly employed in the distribution chains. That means that the freeze-concentrated phase is more or less fluid, and solute mobility may be significant. Frozen storage usually continues for long periods, so products undergo deterioration, which can be as important as those due to the freezing and thawing processes. The underlying reason for necessary extensive studies about change of quality during storage is

**Table 1** Practical Storage Life (PSL) in Months at Several Temperatures

Product	−12 °C	−18 °C	−24 °C
Beef steaks/cuts	8	18	24
Ground beef	6	10	15
Pork steaks/cuts	6	10	15
Fatty fish (glazed)	3	5	>9
Lean fish	4	9	>12
Butter, lactic unsalted	15	18	20
Butter, lactic salted	8	12	14

Source: Adapted from IIR-IIF [1].

**Table 2** Tg<sub>2</sub> Values for Frozen Food Products.<sup>a</sup>

	$Tg_2(^{\circ}\mathrm{C})$	Reference
Lemon juice	-38	Maltini (15)
Orange juice	-34	Moreira (16)
Raspberry juice	-31  to  -35	Moreira (16)
Strawberry	-43	Sa-Sereno (11)
Strawberry juice	-45	Torregiani (17)
Apple	-37	Guegov (18)
	-71	Sa (19)
Tomato	-21	Guegov (18)
Carrot	-32	Guegov (18)
Egg white	-38	Simatos (20)
Egg yolk	-32	Simatos (20)
Beef muscle	-85	Simatos (20)
	$\sim -70$	Sator (21)
Tuna	-70	Inoue (22)
Commercial ice creams	$-27.5 \text{ to } -40^{\text{a}}$	Levine (23)
Commercial ice creams	$-34^{a}$	Blond (24)
Model ice creams	$-25 \text{ to } -43^{\text{a}}$	Hagiwara (25)

<sup>&</sup>lt;sup>a</sup> Depending on the recipe, particularly of the used sugars.

that we do not know enough about the causes of deterioration to be able to propose efficient predictive models.

# II. CHANGES IN FROZEN FOODS DURING STORAGE

The main factors affecting frozen food quality during storage can be divided into two categories: processing and compositional factors. The quality factors associated with processing parameters are mostly related to the ice phase. Defects associated with compositional factors are related to chemical reactions and affect flavor, texture, appearance, color, and nutritional properties.

# A. Physical Changes

### 1. Water (Moisture) Migration

The major physical change that occurs during storage of frozen foods results from water migration. Solid/liquid and especially liquid/crystal transformations can occur with temperature fluctuations, but important water motions could take place without temperature changes. There is always, inside a package or a product, some difference of water vapor pressure due to a temperature gradient or a surface energy difference. As water molecules are not completely immobilized by low temperatures [26], a significant redistribution can be observed in frozen products during the storage time because of the duration usually involved. The water migration causes either water content changes or

changes in ice crystal size called recrystallization. Water migration is highly temperature dependent, and it occurs at any storage temperature, although good conditions (e.g., low temperature, no temperature fluctuations) minimize its importance.

a. Migration with Change in Water Content Water migration can correspond either to a water loss through surface dehydration or to only a water location change for products with heterogeneous water contents.

The surface dehydration adds to dehydration suffered during the freezing process, which can vary from almost zero for packaged products and cryogenic freezing to 3-4% for naked products and poorly designed freezers [27]. These moisture losses have the same origin, i.e., a temperature difference between the product and the surrounding atmosphere, resulting in a difference of water pressure that produces a water molecular flux from the surface of the frozen product to a colder area. Ice sublimation progresses during storage, and it is more pronounced when the storage temperature is higher. A common example is observed with frozen fruits and vegetables that are not protected by transparent films in close contact with them. The films cool faster than the surface of the product, setting up a water pressure gradient; thus a small amount of vapor water migrates from the product to the inside surface of the package. When the freezer temperature increases, the process is reversed and the water vapor condenses on the product surface. The dehydration must be considered irreversible, reabsorption of water in the product being not possible; this removed water crystallizes and remains inside the package as frost. As this cycle is repeated, the buildup of frost in the package becomes noticeable, this effect being amplified by the frequency and magnitude of temperature fluctuations; fluctuations of more than 2°C promote an important sublimation of ice. The consequences are more or less important depending on the products, as the water losses can reduce the weight of the product, thereby reducing the market value for large pieces of meat sold before transformation; overall, the dehydrated surface of a product reduces its appeal to the consumer. Meat appears darker when frozen, since oxygen cannot produce the bright red color of oxymyoglobin (its "natural" color returns on thawing). A surface dehydration on this product leads to the important defect known as freezer burn, particularly for meat carcasses, cuts, and poultry, stored without an adequate packaging. Dehydrated surfaces come in grayish patches, because the disappearance of ice crystals by sublimation forms small cavities on the surface that appear grayish because of the light scattering. Dehydration of unprotected fish can lead to irreversible quality loss when the fish surface becomes dry with a "woody" texture. This dehydration also contributes to increase the rate of rancidity and discoloration. The dehydrated surfaces of vegetables may also appear clearer before thawing, but color changes for these products are predominantly caused by oxidative reactions.

The dehydration effects almost completely disappear on thawing and cooking, except if the product has been stored in bad conditions and dehydration was too severe. But the appearance in the frozen state needs to be taken more into consideration, because it is decisive for the consumer considering whether to buy the product or not. Moreover, the melting of frost during thawing causes an unpleasant appearance when a wet layer appears on the product surface.

Today these defects are more rarely observed, because efficient packaging prevents products from dehydration. Each product to be stored in a freezer over an extended period should be wrapped. Moisture-impermeable films offer considerable protection against moisture loss, but products must be packed with a minimum head space to reduce frost; vacuum packing, which ensures maximum contact of film to product, suppresses all frost and also limits the thermal gradient. Its use is limited because it is more expensive and

unsuitable for delicate products. Surface dehydration can also be limited by coating with a continuous film of ice or glaze. Glazing consists in the application of a potable water spray immediately after freezing, a thin layer of ice covering each piece (fish, shellfish, fruit, vegetable) as protective coating; the water amount is normally in the range of 5 to 10%. Ice glaze is considered to be a cheap means of protection for frozen fish. The glaze is also used for giving some mechanical protection to the delicate frozen florets of cauliflower and broccoli. The formulated coatings, which are added-value processing for fish and meat, are also a protection against dehydration.

More difficult to suppress is the water migration in frozen foods containing regions with large water content differences, generally prepared frozen foods that are a combination of several dissimilar components. There is no particular problem if each component contains ice crystals: the water activity is the same in the whole food since it is only a function of temperature. But when a part of the food has a low water content, and consequently contains no ice, there is a high water pressure gradient between the "dry" part and the ice of the "frozen" part, irrespective of temperature. The resulting water migration produces moisture redistribution. These local water content changes can be important, and detrimental, for the product quality. They are found in frozen pies, fancy cakes, and desserts. Water migrates from the filling to the crust of frozen pies and pizzas or from ice cream to the wafer, and the crispy components become moist and soggy. In frozen bread, if a thermal gradient adds to the difference in moisture, water migrates from the center to the region just under the crust and then freezes to form an icy area, which produces crust separation from the crumb at thawing.

As the water vapor pressure is always higher in the part containing ice, edible barriers with low water permeability must be developed, based on hydrophobic substances such as lipids and waxes, to stop or at least slow down the water transfer. The top of the baked crust of pies or pizzas may be sprayed with shortening to keep the sauce moisture from soaking into the crust. Cocoa butter or chocolate coatings are widely used in the ice cream industry for protecting wafer.

b. Migration Without Water Loss: Recrystallization The quality objectives of the frozen food industry are to set a homogeneous ice crystal distribution and to preserve it during storage. The initial freezing process, particularly when occurring rapidly, yields a highly dispersed crystal phase. This initial distribution promotes metastability: small crystals are thermodynamically less stable than large ones, since they have a higher surface/volume ratio and therefore a higher excess surface free energy. The size and consequently the number of ice crystals must change.

It is not sufficient to produce products with a specified structure through control of the nucleation and crystallization rates. During the storage not only the size and number of ice crystals change but also their shape and orientation. This process is known as recrystallization; it corresponds to water migration as a result of local water motions allowing molecular diffusion from one ice crystal to another, more often without change in ice content. Mazur [28] and then Fennema [29] described several mechanisms of recrystallization; they include migratory, accretive, isomass, and irruptive recrystallizations. Melting and refreezing (regelation) due to temperature fluctuations is also defined as a recrystallization process, which causes the most significant and rapid changes in ice crystal distribution. Except for irruptive crystallization, the mean ice content is not altered by these different mechanisms.

Migratory recrystallization, which is referred to as "grain growth," is also known as the Ostwald ripening. It corresponds to the tendency for the large crystals to grow at the expense of small crystals.

The equilibrium shape of a crystal is the one that minimizes its surface free energy (at constant temperature and volume of crystal); the free energy of a crystal is at a minimum when its structure is perfect and its size infinite. A difference in stability brings about the size difference at constant temperature. The thermodynamic basis is the Kelvin equation, which states that the chemical potential of a curved surface differs from that of a plane one, and relates the vapor pressure over a spherical solid surface to the radius of curvature (r) [28]:

$$\mu_2 - \mu_1 = RT \log \frac{p_2}{p_1} = 2V\sigma \left(\frac{1}{r_2} - \frac{1}{r_1}\right) \tag{1}$$

where  $\mu_2$  and  $\mu_1$  are the chemical potential of the two crystals,  $p_2$  and  $p_1$  their respective vapor pressures,  $r_2$  and  $r_1$  their mean radii, V is the molar volume of ice, and  $\sigma$  the interfacial tension. The relationship between pressure and melting point (the Clausius Clapeyron equation) allows us to calculate the crystal melting point (Tm) as a function of its curvature radius. The undercooling due to the curvature radius decreases as the sphere size increases. For a given storage temperature there is one crystal size that is in equilibrium: a solution with small crystals could also be regarded as being undercooled as compared to the one with large crystals. For pure water, Tm varies from -0.004 to  $-3.99^{\circ}$ C when the crystal radius decreases from 10 to  $10^{-2} \, \mu m$  [30]. Different values can be found in the literature, indeed some parameters are difficult to estimate, particularly the interfacial tension.

Accretion, also termed sintering, is the joining together of two crystals or more to form one larger crystal. A neck is formed between two adjacent crystals, and this neck grows until the original crystals are indistinguishable. The chemical potential, and consequently the vapor pressure, of a spherical surface is greater than that of a concave surface (r is negative). Reducing the surface concavity can lessen this difference in chemical potential. The vapor pressure difference is always the driving force that explains the neck construction. Kingery (cited by Mazur [28]) found that this growth was strongly dependent on temperature: in pure water the neck growth rate decreased from 3.3 to  $0.033 \, \mu \text{m} \cdot \text{s}^{-1}$  when the temperature decreased from  $-2 \text{ to } -25^{\circ}\text{C}$ . In systems where the ice volume is important, this mechanism is certainly dominant, the ice crystal contacts making migration easier. Sutton and coworkers [31] showed that the recrystallization rate was dependent on the ice volume, and the accretion rate decreased as ice phase volume decreased.

Isomass recrystallization: Ice crystals generally tend to become more spherical through isomass rounding during storage even at constant temperature [32]. The driving force is also the water pressure gradient; the Kelvin equation can be used considering different local curvature radii due to the irregular surface. Experimental control has been made in model solutions [33]. In actual foods, the ice crystals are so closely packed that isomass crystallization may not be clearly identified, as water molecules may easily diffuse between different crystals; the end results, however, are identical: all crystals become smoother. It must be emphasized that the transport processes by which a crystal can change its shape at constant volume may be very slow by comparison with those involved in its growth. Theoretically, recrystallization could occur at a significant rate only for crystals with diameters less than about  $2\,\mu m$  [29]. In frozen foods, generally the average crystal size is much greater than  $2\,\mu m$ , but temperature fluctuations enhance recrystallization.

Melt-refreeze recrystallization is observed during temperature oscillations. When the temperature increases, ice crystals partly melt and become smaller; they grow again when the temperature decreases. The smaller ones, which have the highest free energy and the lowest melting point, melt more, grow less, and so gradually decrease in size over time. They can completely disappear; as there is no formation of new nuclei, water refreezes on the surface of remaining crystals, and the total number of crystals decreases and their mean size increases. Fluctuating temperatures stimulate crystal growth; small temperature fluctuations are common during the long life of frozen products, particularly during distribution, but also because of the cyclic nature of refrigeration systems and the need for defrosting. Melt–refreeze recrystallization occurs in combination with other growth modes; most often it is not possible to distinguish which mechanism is the more important. The duration of temperature cycles can also be important for allowing migration of water molecules from one crystal to another; if the temperature fluctuations are fast relative to the diffusion rate of water and/or solute molecules, the crystal changes can be slowed down.

Irruptive recrystallization: Additional ice formation can occur during rewarming; this is referred to as irruptive recrystallization or devitrification. These terms could be discussed because (a) they assume that the medium had already been frozen and in fact they are generally used for systems cooled without or with incomplete crystallization and (b) they should imply that a vitreous state was obtained for the unfrozen medium. Cooling may result in products that are not in equilibrium with respect to the freezing point (Tm) curve. The amount of ice is smaller than predicted; the unfrozen phase is diluted compared to the theoretical equilibrium state. This situation is possible only in case of ultrarapid freezing or for products with high viscosity and/or relatively low water content. This state is stable only at temperatures below the corresponding glass transition temperature (Tg). Above the product crossing the temperature range where crystal nucleation and growth rates are maximum (see Chapter 1), the equilibrium state is obtained by a sudden appearance of a crystallized state.

Most foods, however, contain large amounts of water, and the freezing rates used in industrial processes are sufficiently low to produce a cryoconcentrated phase in thermodynamic equilibrium. This feature could be found in the high pressure assisted freezing process; after pressure release, the nucleation, in the undercooled product, is instantaneous and the crystallization rate is very fast.

Consequences of Crystal Growth In many frozen food products, large crystals have an important adverse effect on the texture. In ice cream and frozen dairy desserts, a coarse texture can result from an increase in the mean size of ice crystals, which become perceptible by consumers. For delicate plant tissues (strawberries) a destruction of cell walls is often observed, which produces a loss of turgidity due to a decrease in the capacity for water retention. Meat and fish are a little less sensitive to ice crystal size changes, but crystal growth might enhance shrinkage of muscle fibers and even disrupt the cellular structure, resulting in a greater chance of oxidative catalysts coming into contact with reactive components. In view of the importance of ice crystals in modifying the texture of frozen foods, it is not surprising that there have been considerable efforts to control ice crystal size, and scientific investigations to bring to light the parameters that play on the different recrystallization modes. The higher the storage temperature, the faster is the crystal growth. The influence of the storage temperature is well documented: for example, the mean ice crystal diameter, which was of  $10 \,\mu m$  in beef muscle frozen at  $-40^{\circ}$ C, becomes equal to  $40 \,\mu m$  after a 150 h storage at  $-5^{\circ} \text{C}$  [34]. This crystal growth lead to greater tissue disruption, protein denaturation, and exudate production [35].

Crystal size has considerable importance in ice cream manufacture; ice cream is the only frozen product consumed in the frozen state, and large ice crystals make the product appear colder and sandy. Polysaccharides were reported as efficient stabilizers in several ice cream studies; they alter both initial crystal size and recrystallization rate [36]. In a study on the cryoconcentration process, Smith and Schwartzberg [37] observed that the presence of gelatin resulted in a decrease in ripening rates. The reported effects of polysaccharides on the slowing down of recrystallization are real; but the mechanisms by which they exert such effects are not well understood. Among the potential mechanisms that have been suggested are an effect on viscosity of the cryoconcentrated phase [10], and the polymer adsorption on ice crystals [38]. If the formation of a weak gel slows down the initial crystallization rate [39,40] by a hindering mechanism, the water diffusion that allows the recrystallization is not slowed down by the existence of a network [41].

There is no evidence to support the view that polysaccharides could affect the amount of ice formed, which is in agreement with the thermodynamic properties of solutions. They can even enhance the process of nucleation by the introduction of specks (outside impurities) [42].

# 2. Solute Crystallization

Many solutes are supersaturated in the unfrozen phase, particularly sugars. After a while, they may crystallize, as is the case for lactose in ice cream. Even if the lactose crystal size is smaller than that of ice crystals they give a sandy texture because they melt more slowly than ice crystals in the mouth. This effect is limited by polysaccharide addition, but an important lactoprotein content increases its risk if lactose proportion becomes large. Sugars may also crystallize on the surface of high sugar fruit spreads during frozen storage, giving the appearance of mold growth on the surface. The same effect can be observed with icing or glazes of frozen desserts; the soft texture of these latter must be preserved by a modification in formulation: replacement of sucrose by invert sugar and addition of a stabilizer such as gum arabic. The formulations used must have the greatest degree of tolerance to temperature abuse. While a certain amount of temperature fluctuation is unavoidable, efforts should be made to minimize it.

In most cases, the texture quality of frozen food products is better preserved when their temperature is maintained as low as practicable during storage. The temperatures used to monitor recrystallization rates are generally high compared to the actual storage temperatures. At -18 to  $-20^{\circ}$ C, the recommended temperatures, crystal growth is much slowed, and the main problem remains the temperature oscillations that induce the melt–refreeze process. This leads us to adopt lower storage temperatures (-25 to  $-30^{\circ}$ C) for long-term storage of frozen finished products awaiting distribution.

### B. Chemical Changes

An abundance of chemical and biochemical reactions may take place in animal and plant tissues, even in dead cells. If during the freezing process most reactions are stopped, some may be only reduced. As a frozen product is typically available after several months in storage, some changes can be observed thus far, but they will be minimal if optimal conditions of storage are respected, i.e., low and stable storage temperature. Chemical reactions are slowed down by the temperature effect, but small solutes retain some mobility in the unfrozen phase. Because of freeze-concentration, reactants are in close proximity, and reactions rates may increase. It has been shown that freeze-concentration is

likely to increase reaction rates over the subzero temperatures, the temperature range depending on substrate composition [43,44]. At lower temperatures, reactions may become diffusion controlled and the rates decrease as the maximal freeze-concentration, i.e., Tg', is approached.

Evaluation of the chemical changes during frozen storage is a difficult task: (a) because of the duration of the experiments; at  $-18^{\circ}$ C they have to spread over one or two years; (b) the difficulty of dissociating storage influence from other processes effects: blanching, freezing, and thawing; and (c) because of the diverse nature of the sensitive compounds; the chemical heterogeneity should require experiments for each vitamin, lipid and protein.

Apart from the general problem of analysis methods, there are many possible sources of variations in product quality: for animals, breed, age, diet, preslaughter treatment; for plants, cultivar, maturity, harvesting practice; and recipes for prepared foods. The complexity, variability, and diversity of interactions between different ingredients, and thus the physical laws that control their reactions, are unknown. This may also be due to incomplete destruction of enzymes during blanching or cooking, or to oxidations permitted by packaging materials permeable to oxygen.

The analyses done at different temperatures allow the determination of the reactions  $Q_{10}$ , i.e., the ratio of deterioration rates at two temperatures separated by a 10°C interval; it indicates how much longer the product quality will be retained when the temperature is decreased by 10°C. The  $Q_{10}$  values can be high for frozen products, varying from 2 to 30, while for temperatures above 0°C they are generally between 2 and 3.5.

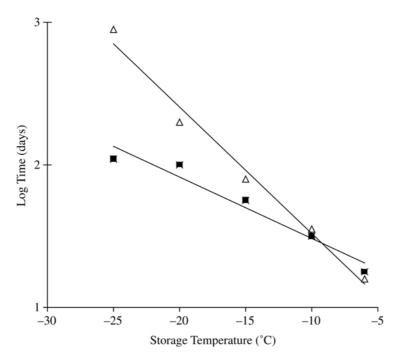
This complexity can be illustrated by a comparison of chlorophyll degradation in frozen green beans either blanched or unblanched [45]. The rate of color degradation was faster in blanched than in unblanched green beans at  $-6^{\circ}$ C, while the opposite was observed at  $-18^{\circ}$ C. This study well demonstrated that comparison at only one temperature can be misleading; this can be explained by the difference in  $Q_{10}$  values, respectively equal to 7.8 and 2.8 at these temperatures (Fig. 4). The degradation ways are different: in unblanched green beans, the degradation is associated with the peroxidation of some fatty acids by lipoxygenase, which gives uncolored substances; in blanched green beans, the bright green chlorophyll is converted to olive green pheophytins, independently of enzymes and oxygen presence.

The difficulty of analysis and of interpretation is also demonstrated by the review by James and Evans [46] on the storage life of beef, pork, and lamb at different temperatures. Experimental data from many publications show there is agreement that storage temperature influences frozen state life, and that in general the lower the temperature the longer the shelf life. But considerable scatter between results at any one temperature is observed. Moreover, few papers present data at different storage temperatures, and many experiments are realized at too high a temperature compared to storage common practice.

Different types of chemical changes are particularly troublesome: oxidative processes and protein degradation, denaturation/aggregation, result in texture, flavor, and color degradations, and vitamins losses, i.e., decreases in sensory and nutritional qualities.

### 1. Oxidative Deterioration

Lipid oxidation corresponds to the most important chemical reactions associated with quality change during frozen storage. In foods, lipids can be oxidized by both enzymatic and nonenzymatic mechanisms. It is generally agreed that autoxidation, that is, the reaction with molecular oxygen, is the main reaction involved in oxidative deterioration of



**Figure 4** Days of storage required for a 10% decrease in chlorophyll of blanched ( $\triangle$ : r = -0.99; Q10 = 7.8) and unblanched ( $\blacksquare r = -0.97$ ; Q10 = 2.8) frozen green beans (var. Koba) as a function of temperature. (Redrawn from Ref. 45.)

lipids. The major pathway for oxidation involves a self-catalytic free radical mechanism, which can be presented as a three-step simplified scheme: (a) initiation in which free radicals  $R^{\circ}$  are formed from unsaturated lipid molecules RH by their interaction with oxygen in the presence of catalysts; (b) propagation in which the free radicals continue to be generated, and (c) termination when an antioxidant, an oxygen or free radical scavenger, reacts with free radicals. Lipases and phospholipases are enzymes, which hydrolyze lipids, the release of fatty acids can lead to rancid flavor. Lipoxygenases have also proved to be active in frozen foods and to cause changes in lipid constituents. This does not only hold for animal tissues, rich in lipids, but also for plant products. Lipoxygenase is mainly responsible for the deterioration of flavor occurring in frozen unblanched vegetables.

Lipid oxidation most commonly occurs in fatty meats and is considered as the major cause of frozen shelf life reduction compared to lean meat species. The importance of lipid oxidation varies with the quantity and the nature of the lipids; highly unsaturated lipids are less stable than saturated ones. Polyunsaturated fatty acids are autoxidized in the presence of oxygen to hydroperoxides that decompose into volatile compounds, forming flavor and aroma compounds characteristic of rancid foods.

Color degradation is also related to oxidation during storage. For example, the discoloration of tuna is due to the oxidation of myoglobin to metamyoglobin; for salmon, the pink color, given by carotenoids, can disappear by oxidation after a long storage. The browning of fruits is due to the action of polyphenoloxidases and molecular oxygen on colorless phenolic substances. Fatty acids formed during autoxidation may produce indirect effects on textural degradation by promoting protein denaturation [47].

For animal tissues, the stability studies focus on lipid oxidation. Clearly, composition has an appreciable effect on the storage stability of frozen products. Fish products are the best example, their shelf life being related to their fat content: fatty fishes have a shelf life of 4–6 months at  $-18^{\circ}$ C, whereas the lean ones can be preserved 7–12 months [48]. Pork meat fat contains a higher proportion of unsaturated fatty acids than other meat fats such as beef, lamb, and chicken; consequently pork has a shorter storage life (Table 1). Antioxidants are regularly used to stabilize the flavor of composed foods; they delay the development of rancidity by interfering with the initial step of the free radical reactions or by interrupting propagation of the free radical chain [49]. Antioxidants are of little effect in intact muscle; they are more effective when mixed with ground meats; their efficiency appears better when used as nutritional supplements. Also, the very undesirable effect of salt in flesh products is well noticed [50].

For vegetables and fruits, color changes are predominantly caused by oxidative reactions. Enzymatic browning is due to polyphenoloxidase promoting the direct reaction of polyphenolic substances with air oxygen. This problem can be overcome by blanching, but fruits generally are not blanched, because this process gives them a cooked flavor and a soft texture. Color stability is improved by the presence of ascorbic acid or the addition of citric acid, which maintain the phenolic substances in a reduced colorless state. Sugar or syrups added to fruits before freezing also help to limit browning by slowing down the enzymes' action and by protecting against oxygen. The stability studies focus on vitamin changes over time. Vitamin C (ascorbic acid) is the vitamin most readily associated with vegetables; it is also the most labile vitamin. It is oxidized by oxygen, the phenomenon being enhanced by active oxidizes (ascorbases, catecholoxidases) and metallic elements (Fe, Cu). Vitamin C content varies considerably between different vegetables, but also in a particular vegetable, depending upon variety, maturity, and agronomy. The level of vitamin C is not an indicator per se, but this latter is generally used as the most appropriate "marker" for monitoring quality changes during processes. Favell [51] compared the vitamin C content in some commercially quick-frozen vegetables stored for up to 12 months with the same fresh vegetables at various stages of distribution. For frozen products the major loss attributed to pretreatments; changes observed after storage at -18°C for 12 months were minimal, and the frozen product presented the best nutritional qualities among products usually available to the consumer. This report confirms that for all frozen vegetables, nutritional quality is equal to and even better than that of fresh vegetables bought in supermarkets; frozen vegetables are also "fresher" than those preserved by other thermal processes.

### 2. Protein Denaturation

The conformation of protein derives from its secondary and tertiary structure. As a result, every treatment of proteins with concentrated saline solutions, organic solvent, heat, and cold may modify this conformation. Denaturation is an elaborated phenomenon during which new conformations appear. The effects of protein denaturation are numerous: decreased solubility, altered water binding capacity, loss of biological activity, particularly enzymatic, and increased susceptibility to attack by proteases due to the unmasking of peptide bonds in unfolded structures. The sensitivity of a protein to denaturation is related to the readiness with which the denaturing agent breaks the stabilizing interactions. Since the latter vary according to the protein, the resulting effect of the same treatment will be variable. The toughening and poor water retention observed in some species of fish, the precipitation of casein micelles in milk, and the gelation of egg yolk lipoproteins result

from a decrease in protein—water hydrogen bonding and an increase in protein—protein interactions in the frozen product. The myofibrillar proteins, which aggregate during frozen storage, are probably linked by secondary interactions and disulfide bonds. As these aggregates tend to grow in number and size, the proteins lose more or less of their water-binding capacity.

Freezing concentrates solutes, including salts and small organic molecules. Changes in ionic strength and possibly pH [52] in the remaining unfrozen aqueous phase ensue, leading to the denaturation of proteins. Aggregation and precipitation may occur. The most popular tests to determine the denaturation during storage of frozen meat or fish, for example, are the determination of the loss in solubility or extractability and the measurement of the water-retention properties of the muscle system.

The use of cryoprotectants in cured and processed meats can stabilize the functional properties of myofibrillar proteins; for example, addition of polyhydroxy compounds such as sorbitol and sugar in comminuted fish flesh allows us to preserve the gel-forming ability essential to obtain a surimi pasta.

Even at the low temperatures used for storage, most enzyme systems are still active. Quality deterioration is most commonly observed in meat and fish products because they are raw materials, whereas, vegetables are generally subject to blanching, which destroys most enzymes, before freezing. However, the high concentration of the unfrozen phase increases the possibility of contacts between residual enzymes and substrates. Since some proteins also act as enzymes, attempts have been made to assess denaturation from the angle of enzyme inactivation. The loss of protein activity is more reflected by enzymatic activities than by extractability or solubility. It is possible that enzymatic reactions are accelerated during freezing because of the cofactors' concentration and because of membrane disruption, which allows easier enzyme–substrate interaction than in intact cells.

# C. Microbial Processes

Frozen food products and particularly fish products are far from sterile and cannot be considered as microbial safe products. Many microorganisms are not destroyed by the freezing process and may survive even if they remain inactive during storage.

The effects of frozen storage on microorganisms are variable, depending upon the organism considered. The lower limit for bacterial growth in food is about  $-10^{\circ}$ C and for yeasts it is about  $5^{\circ}$ C lower. However, it is not possible to store food for long periods at temperatures slightly below the growth limits of microorganisms. Foods thus stored show noticeable signs of deterioration that should be also due to the activity of microorganism enzymes, adding to that of the product specific enzymes. Whereas a heat treatment used before freezing decreases the number of surviving microorganisms, the enzymes secreted into the food, such as lipases and proteases, are much less affected by heat.

At  $-18^{\circ}$ C no growth of microorganisms will occur. The reduction in number owing to storage is possible but not of practical importance. Therefore the quality of raw products and a good hygiene during the production are most important.

In the practical application of freezing technology, microbial processes are essentially limited to the effects of microbial enzymes; frozen products with a maximum shelf life can only be produced when raw materials of initially outstanding quality are used [53].

### III. STABILITY AND SHELF LIFE

# A. Which Model for Stability Studies in Frozen Foods?

The principal factors affecting the quality of frozen foods during their storage are (a) storage temperature and time, (b) nature and quality of the product at the time of freezing, (c) preparation and freezing processes, and (d) packaging.

The first two are referred to as TTT factors (time-temperature tolerance). They have been widely analyzed during the 1950s and 1960s and have allowed important progress in frozen food quality [54]. The two following decades have considered the PPP factors (product, process, packaging) [50]; it has been clearly proved that frozen product quality is directly related to the quality of the raw material used and to the optimization of preparation processes (heat treatment); moreover good packaging may often more than double their storage life.

Today the studies' objectives are to elaborate predictive models based on a more accurate knowledge of physical state and molecular mobility as a function of the storage temperature.

Levine and Slade [2] have promoted the idea that the transition of a liquid to a glassy state for the maximally freeze-concentrated fraction, and consequently the temperature at which this glass transition takes place (Tg'), was the threshold of instability, and that the kinetics above this temperature were controlled by the difference between the storage temperature and Tg'. It is essential however, to define a shelf life for each frozen food product on the market. Product shelf life can be estimated by (a) objective measurements of properties or characteristic changes related to food quality, e.g., the ascorbic acid content of frozen fruits, and (b) shelf life failure criteria based on some noticeable quality difference between experimental and control samples.

Around 1960, it was suggested that in regulations pertaining to the keeping quality of a frozen product one should use the concept of time first to *just noticeable difference* (JND). In IIR-IIF publications [1] this is referred to as a product's *high quality life* (HQL). HQL is defined as the elapsed time between the freezing of a high-quality product and the moment when 70% of an experienced taste panel is able to distinguish, by triangle tests, the product from the control stored at a very low temperature ( $< -40^{\circ}$ C) [54]. Sensory quality can also be estimated using an hedonic scale method. But obviously consumers are not as sensitive to a small change as is a taste panel, and an acceptability time has been introduced that corresponds to the concept of *practical storage life* (PSL). It corresponds to the consensus of opinions about the useful "storage life" to be reasonably expected for the product.

There are many occasions for frozen products to suffer heat shock abuse during distribution and to reach an unacceptable level of quality. Since there is no way of identifying the conditions under which frozen products will be handled once they leave the manufacturer's control, it is problematic to predict an exact shelf life for any frozen product. A practical approach can be based on the profile of received consumer complaints, combined with the evaluation of products purchased at or near the sell by date. The level of complaints involving chosen quality attributes in most aged products would support a decision to extend or reduce the sell by date.

Thus the shelf life of a product essentially becomes a measure of the suitability (or lack of suitability) of a product for consumption.

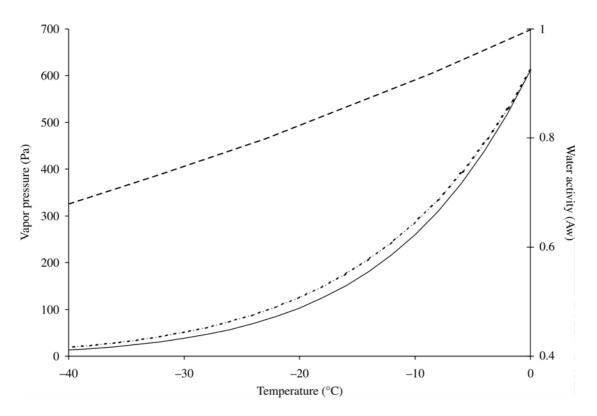
No precise relationship has been demonstrated between HQL and PSL, the time gap measured between the two varying with the product. Moreover, the TTT relationships are not mathematical functions. The TTT relationships are deduced from experimental

studies, and the data can only be compared so long as the PPP factors are the same. The books by Van Arsdel and coauthors [55], Jul [50], and IIR-IIF [1] reported numerous results of storage investigations on the keeping quality of frozen foods and PSL and HQL data.

# B. Glass Transition and Stability Prediction

### 1. Vitreous State in Frozen Materials

As described before, all physical, chemical, and biochemical changes occurring during the storage of frozen foods are strongly affected by temperature, and also by water availability; the temperature effect is associated with the increasing concentration of the liquid phase as the temperature is lowered. Comprehensive procedures have been searched for accurately predicting stability. Temperature is of little value; the shelf life of different products is not the same at a given temperature. Water activity (Aw) is also not effective as a predicting parameter. The freeze-concentrated phase being in equilibrium with ice, its partial water vapor pressure is equal to that of ice at the same temperature. Thus for all frozen foods the same water activity would be expected at the same temperature; the water activity in frozen foods only depends on temperature (Fig. 5). Levine and Slade [2] proposed that the changes in amorphous concentrated systems should be controlled by kinetic rather than thermodynamic constraints. Consequently, the viscosity and other relaxation properties would be appropriate to predict the rate of reactions when molecular mobility is reduced.



**Figure 5** Water activity (Aw) of ice as a function of temperature. Vapor pressure of ice: full line, of liquid water: dotted line, Aw: dashed line.

When temperature decreases, diffusion-controlled processes slow down with the increase in viscosity due to the combined effects of concentration and temperature. In the glassy state, i.e., below Tg', chemical reactions should be strongly limited, and a long time stability expected. In practice, foods are rarely maintained at storage temperatures below Tg'; as described before, they become vulnerable to crystal ripening and chemical changes.

The shelf life of frozen food products should be largely controlled by the physical state of their freeze-concentrated fraction; then, the properties and behavior of the unfrozen phase appear to be the key factor for understanding and improving the stability of frozen foods and their shelf life.

The glass-liquid transition is characterized by a rather abrupt change in many physical properties of the material. The main observable phenomenon is a change in the mechanical properties: the glassy brittle solid is changed into a "rubber" in the case of a polymeric material, or in a viscous liquid the viscosity of which strongly decreases as the temperature increases. The most important implication of the glass transition concept in frozen foods, and foods in general, is that translational motions of molecules, especially larger ones, do not occur in the glassy state during a practical time frame. Storage at a temperature above this characteristic glass transition range would result in pronounced changes in mobility of solutes and an increase of degradation rates. There should be two possibilities for improving the stability of a frozen product: to lower the storage temperature below its Tg' and to modify the latter by a recipe change, a possibility for manufactured foods.

However, experimental data that analyze quantitatively the relationship between the stability of actual frozen foods and the glass transition are very scarce, and the few tests [56,25,57] are not always conclusive. This scarcity cannot be attributed to a lack of interest in this parameter but rather to the credibility of data as found in the literature. The published Tg' values for sucrose frozen solutions, as collected by Sahagian and Goff [6], vary from -32 to  $-46^{\circ}$ C, and even their designation is different: Tg', T'm,  $Tg_2$ , and also  $Tg_{r2}$ ; for fish the values vary from -70 [22] to  $-13^{\circ}$ C [58]. There is general agreement on the concept, but it is necessary to clarify the important discrepancies as regards its practice. The question is not so much what is the true value of Tg' but more about the molecular mobility changes in that temperature range. Two aspects must be considered: (a) to have reliable information on the glass transition temperature of the maximally freeze-concentrated phase (Tg'), and (b) to know in which conditions Tg' is the reference temperature.

## 2. Influence of Composition on Tg'

For a series of homologous polymers it is well known that the temperature of the glass transition is increasing with the molecular weight; the same is shown to be true for sugars [59]. Other molecular properties such as branching [60] and cross-linking [61], play a lesser role. For a mixture, the glass transition temperature can be predicted from the characteristics of each component by means of expressions such as the semitheoretical Couchman and Karasz expression [62]:

$$\ln Tg = \frac{\sum_{1}^{n} (C_i \Delta C p_i \ln Tg_i)}{\sum_{1}^{n} (C_i \Delta C p_i)}$$
(2)

where  $C_i$  is the mass fraction,  $Tg_i$  the glass transition temperature, and  $\Delta Cp_i$  the increment in heat capacity at  $Tg_i$  for each component.

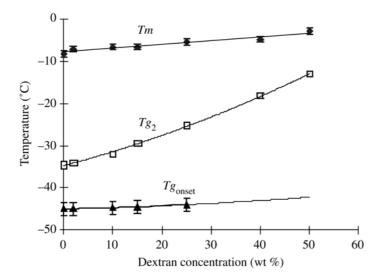
However, given the uncertainties in the  $Tg_i$  and  $Cp_i$  values for biopolymers, and the poor agreement with experimental data when one component is water, the more empirical Gordon–Taylor equation is preferred [63]:

$$Tg = \frac{C_{\rm s}Tg_{\rm s} + kC_{\rm w}Tg_{\rm w}}{C_{\rm s} + kC_{\rm w}} \tag{3}$$

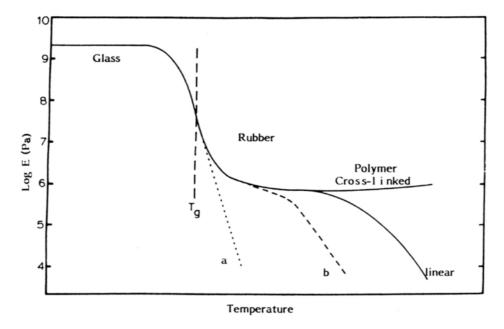
where  $C_s$  and  $C_w$  are the mass fractions of solid and water,  $Tg_s$  and  $Tg_w$  their Tg values, and k a fitting parameter.

The thermodynamic properties of solutions depend on the size and nature of the solute; thus composition changes modify both the Tm and Tg curves, and consequently the Tg' value. Tg' will be influenced mainly by the molecular weight (MW) of the solute and to a lesser extent by its conformation and structure; for example, Tg' is around  $-40^{\circ}$ C for sucrose solutions and  $-46^{\circ}$ C for glucose solutions.

The presence of high MW solutes could increase the Tg' and could be expected to improve stability. Hydrocolloids (polysaccharides or gelatin) must be added at a rather high concentration to a solution of low MW solutes, to have a significant impact on Tg'. In fact, they essentially alter the second step  $(Tg_2)$  of the transition;  $Tg_{\text{onset}}$  and  $Tg_1$  do not increase (Fig. 6) [64]. This may indicate that the temperature at which some mobility appears is not raised upon the addition of the polymer. At the opposite, low MW solutes have a plasticizing effect and thus lower the Tg' of multicomponent samples. The addition of mineral salts largely decreases Tg'. For frozen saline sucrose solution containing 26% sucrose +6% NaCl, Tg' decreases to -75°C [65]; with CaCl<sub>2</sub> in the same ratio, Tg' equals  $-90^{\circ}$ C [66]. The Tg' of fish at  $-70^{\circ}$ C can be explained by the presence of salts in the flesh composition [22]; this low-temperature transition is confirmed by mechanical measurements [67]. Chang and Randall [66] showed that if for frozen pure protein solutions the Tg'is high  $(-10, -12^{\circ}\text{C})$ , it decreases with the addition of small solutes (sugars, polyols). It is a common observation that in mixed materials, the glass transition range broadens; for instance, with a mixture of salts, the transition may spread over a range of nearly 30°C [68]. The presence of ice may promote this broadening, amplifying the  $Tg_2$  feature supposed to be due to enthalpy relaxation associated with the beginning of ice melting.



**Figure 6** DSC transition temperatures as function of composition for frozen sucrose dextran solutions (50% w/w).



**Figure 7** Theoretical thermomechanical spectra (storage modulus E') versus temperature for different molecular weight components in the glass transition range. (a = low MW solute, b = frozen sucrose + polymer solution.)

Low Tg' values are not always reported for actual frozen food products (meat, fish) because experimental works may not extend to a low enough temperature range for observing the beginning of the glass transition feature.

The addition of hydrocolloids greatly increases the viscoelastic modulus of the freeze-concentrated phase just above Tg'. Mechanical measurements show a tendency toward the development of a rubbery plateau (Fig. 7); this effect increases in the order of increasing shear thinning behavior of polysaccharides [10]. These properties are certainly more representative of the macroscopic properties than of the molecular ones. They depend, moreover, on the behavior of both coexisting phases: the ice crystals and the freeze-concentrated phase. In the latter, the freeze-concentrated polymers are above their critical concentration for entanglement.

### 3. Molecular Mobility Around the Glass Transition and Consequence for Stability

The stability of frozen foods strongly depends upon the storage temperature. This temperature dependence is due to the dramatic decrease in viscosity at T > Tg'. It was tempting to explain the drastic effect of temperature by WLF kinetics [69], the WLF equation [Eq. (4)] specifying a much greater temperature dependence of molecular viscosity and viscosity-dependent properties than the Arrhenius equation [Eq. (5)].

$$\log(\frac{\eta_{\rm T}}{\eta_{\rm Tg}}) = \frac{C_{\rm 1g}(T - Tg)}{C_{\rm 2g} + (T - Tg)} \tag{4}$$

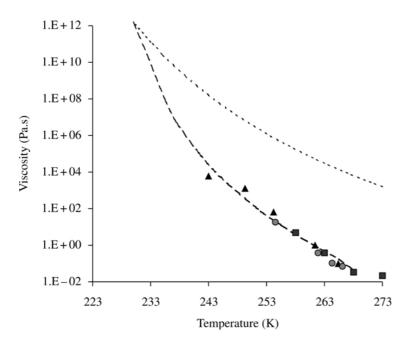
$$\eta_T = \eta_0 \exp\left(\frac{-Ea}{RT}\right) \tag{5}$$

where  $\eta_T$  and  $\eta_{Tg}$  are viscosity at T and Tg, respectively, Ea activation energy, R gas constant,  $\eta_0$ ,  $C_{1g}$  and  $C_{2g}$  are phenomenological coefficients.

Note that the evolution of molecular mobility above the glass transition temperature may be different depending on the material, as shown by the strong/fragile classification of Angell and coworkers [70,71]. Strong liquids are those with Arrhenius or near-Arrhenius behavior above glass transition, in contrast, fragile liquids show an important increase in mobility with increasing temperature.

The major complication of the WLF theory as applied to frozen systems is that the composition of the unfrozen solution changes as the temperature increases owing to ice melting [56]. Thus dilution of the unfrozen solution results in an even greater decrease in mobility than predicted by the WLF equation where Tg' is taken as the glass transition temperature (Fig. 8).

In a glass, long range motions are restricted; motions (reorientation of small groups of atoms) are mainly local. The temperature dependence of dynamic properties is generally considered to obey the Arrhenius law [75]. In maximally frozen systems, ice seems to promote structural relaxations in the glass over a broad temperature range below Tg', which may explain the complex features that are observed with frozen sugar solutions by DSC analysis; a slowing down of enthalpy relaxation processes is induced by polymer addition [10].



**Figure 8** Viscosity of freeze-concentrated sucrose solutions as function of temperature. Symbols are experimental values:  $\blacksquare$  (72),  $\blacktriangle$  (73),  $\blacksquare$  (74). Dashed line: the WLF prediction of viscosity. Dotted line: the viscosity predicted for a 82% sucrose solution (maximum cryoconcentration admitted as constant).

# C. Storage Temperature (Ts) and Stability

For products stored at a temperature Ts < Tg', long-term stability may be expected. Translational molecular motions of glass-forming molecules are effectively inhibited; the diffusion-controlled processes cannot be observed on a practical time scale. By contrast, in the product stored at Ts between Tg' and Tm, the mobility of reactive solutes is higher, resulting in losses of nutrients and quality.

The relevance of Tg' as a reference temperature, for predicting the rate of physical and chemical changes during storage in the frozen state, presents no clear bases, although it has often been mentioned. Different experimental data, essentially the rate of ice crystal growth and some enzymatic reactions, have been analyzed with WLF kinetic equation. Sutton and coworkers [76] showed that ice crystallization rates in fructose solutions increased exponentially with increasing temperatures and followed WLF rather than Arrhenius kinetics, but the authors stressed the point that the range and the number of tested temperatures were too small for exact determination of the best model of temperature dependence. The possibility of inhibiting the recrystallization in ice creams by raising Tg' through the addition of high MW compounds was explored in many studies. There was a general trend where the recrystallization rate increased with increasing (Ts - Tg'), but the data did not fit WLF kinetics well [25]. For these models, the reference temperature was taken as constant (Tg'), although the composition of the amorphous phase was variable in the tested temperature range. From a practical point of view, the high MW solutes used as stabilizers in ice creams are added at too low levels (0.1–0.3%) for having significant impact on Tg' of ice cream mix [25], but they certainly slow down the ice recrystallization.

The main mechanism of the beneficial effect of the stabilizer addition to ice cream texture should be related to the perception in the mouth [77].

Manzocco and coworkers studied polyphenoloxidase and peroxidase activity in media with marked differences in viscosity at equal subfreezing temperatures; the rate values for the catalyzed reactions were within the same order of magnitude, in spite of the large differences in viscosity; and Arrhenius kinetics gave a fit at least as good as WLF kinetics over the temperature range of -30 to  $-5^{\circ}$ C [78]. Biliaderis and coworkers [79] presented similar results for the temperature dependence of the oxidation rate of ascorbic acid in the presence of starch hydrolyzates.

More generally, the temperature dependence of many deterioration processes is much too weak to be correlated with the drastic decrease in viscosity above Tg' resulting from ice melting associated with the WLF effect when the latter is calculated using a constant Tg' [80].

For the glass transition concept to be relevant for predicting the rate of diffusion-controlled reactions, several requirements have to be met: diffusion and viscosity should be linked according to the Stokes–Einstein relation, and viscosity should have a WLF temperature dependence; for frozen products, an appropriate glass transition temperature has to be considered, taking into account the varying concentrations in the prevailing storage conditions.

Appropriate WLF parameters  $C_1$  and  $C_2$  have to be used [81], and Tg values much lower than Tg' have to be considered. For frozen food stored above Tg' (Table 2), the freeze-concentrated phase is more diluted than at Tg'; this latter parameter cannot be used as a reference temperature [82]. For example, in ice creams, the Tg' value is close to  $-34^{\circ}C$ ; if the storage temperature is  $-20^{\circ}C$  (Ts), the freeze-concentrated fraction is liquid. Because its state diagram can be compared with that of sucrose–water binary, the

reference Tg to consider in the WLF equation to predict the influence of temperature can be estimated at around  $-75^{\circ}$ C, i.e., the temperature at which the liquid phase would turn into a glass if it could be cooled without further crystallization. The mobility and the resulting stability in the matrix would be related to the difference between Ts and Tgs and not between Ts and Tg' (see Fig. 3).

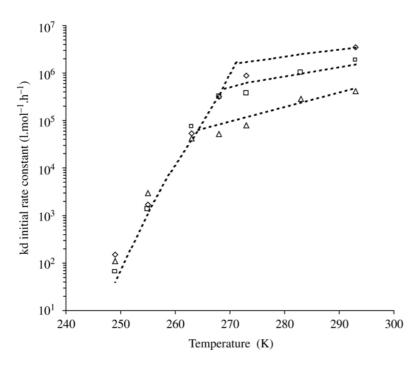
Figure 9 shows that a good correlation can be found between the experimental and the calculated data for the rate constant of the hydrolysis of disodium-p-nitrophenol catalyzed by alkaline phosphatase in sucrose solutions in the  $[+20, -24^{\circ}C]$  temperature interval [72]. The prediction assumes that the reaction rate is viscosity dependent; in the studied temperature range, translational diffusion (D) of reactants and viscosity  $(\eta)$  were found to be linked by the Stokes–Einstein relation [83]:

$$D = \frac{KT}{6\pi r\eta}$$

where r = hydrodynamic radius of the diffusing molecule.

The prediction is based on the measured and calculated viscosity; the WLF equation was used to predict the medium viscosity taking as temperature reference the glass transition temperature Tgs corresponding to the concentration at the storage temperature Ts (Fig. 3).

Even if reaction rates are viscosity dependent, this parameter is not the only one that should be taken into account to predict the complex changes during low temperature storage [78]. A simple relationship, stability = f(Ts - Tgs), cannot be expected; the kinetics of physical and chemical processes taking place in frozen products stored above Tg' may be controlled by many factors depending or not on the concentration changes:



**Figure 9** Initial reaction rate constant kd of alkaline phosphatase as a function of temperature in sucrose solutions ( $\diamondsuit$  30% sucrose,  $\Box$  40%,  $\triangle$  57.5%). Dotted lines:  $k_d$  predicted from the measured viscosity (samples without ice) or from the predicted viscosity for the freeze-concentrated phases. (From Ref 72.)

solute concentration, pH, ionic strength. In structured products cell membranes limiting the solute diffusion, aggregation, and conformational change of protein or enzyme with temperature must also be taken into account. Sometimes enzyme activity is better preserved in model solutions than in the food product [84].

The discrepancy between theory and experiment can also be explained by the difficulty of knowing if, in the temperature range considered, the reactions are diffusion-controlled or still activation-controlled or even in a transition region where activation and diffusion constraints coexist. A more important point is that the molecular diffusion of some components might be influenced by temperature and water content in a way very different from macroscopic properties. When the size of diffusing molecules is very small compared with the matrix molecules, the macroscopic viscosity may not reflect the "local" viscosity and is not the parameter that controls diffusion.

Despite the limitation of the glass transition concept with regard to quantitative prediction of reaction kinetics, the Tg' value of a food product is of practical significance because it should provide a guideline to the industry in the formulation of manufactured foods.

A good stability being related to the possibility of maintaining the product below Tg', the formulation change could be a possible improvement. Attention must be given to the presence of small solutes, which contribute to providing some mobility at low temperatures. For example, the experiments of Wang and Jane [85] show that the addition of sugar increases the starch degradation, even for storage temperatures below the apparent Tg'.

It should also be remembered that storage in the glassy state does not guarantee long-term stability, as this state appears to have only a weak direct impact on the diffusivity of small molecules such as oxygen and water. The relatively high mobility of oxygen in glassy matrixes is often responsible for the oxidation in frozen foods stored below Tg' and thus for their limited shelf life.

Tg' has been proposed as the temperature of critical importance. This parameter represents a limit interesting to characterize the product stability, but it cannot be reliably used as a reference temperature for evolution kinetics above Tg'. The relevant temperature is then the glass transition temperature Tgs of the analyzed medium at the storage temperature.

### IV. CONDITIONS OF BETTER FROZEN STORAGE

How long can a food be maintained in a state of satisfactory quality during frozen storage depends on the type of food, how it is processed and packaged before freezing, and on the storage temperature and how it is handled during the storage time.

Exposure to high temperature and/or fluctuations of storage temperature produces cumulative adverse effects on the quality of stored foods, which are the primary cause of damage to food marketed through retail channels. The rate of quality deterioration increases with increase in storage temperature, and such changes (reactions) are irreversible. If microorganisms will not grow in frozen foods, chemical and physical changes occur at rates that are of commercial significance during insufficiently controlled storage. Violations of recommended storage conditions should be detected and corrections instituted.

Many investigations have been made to control temperature rise in the logistic frozen food chain; these studies show large variations of product temperature even after a

short exposition to ambient conditions. The temperature of the most exposed packaged product in a pallet can exceed  $-15^{\circ}$ C at core for an ambient temperature of  $21^{\circ}$ C over a 25 min period; the surface temperature was 3 to  $5^{\circ}$ C higher than the core temperature. Today mathematical models become more precise and allow us to calculate the consequence of this temperature rise; it should be possible to specify what the initial temperature of the product must be to maintain  $-18^{\circ}$ C throughout the distribution chain [86].

Time-temperature tolerance (TTT) applied to cases or pallets could be of great help in this regard; but they are very little used yet.

# A. Time-Temperature Indicators

Considerable research has been conducted to develop and design reliable systems to monitor temperature abuses. There exist different indicator kinds: temperature indicators showing only exposure above a reference temperature and temperature—time indicators taking into account the cumulative effects of time and temperature above the reference temperature; the latter would be preferred because both temperature and time are important for quality control of frozen foods during storage and transportation.

Various types of time-temperature indicators (TTI) have been developed that are based on mechanical, chemical, microbial, or enzymatic irreversible reactions for visualization of the degree of food quality change [87,88,89]. The problem is that for accurately monitoring changes in frozen food quality the TTI activation energy should be closely matched to that of the food quality factor. To meet such a requirement is a difficult challenge; moreover, an important limit is the cost of such indicators, which should be placed on the outside of each package.

Temperature management requires monitoring devices; today, recording thermometers are available and must be used to monitor distribution conditions.

Samples must also be protected against light for preserving the color stability. Exposure to the levels of light found in some retail frozen food display areas can cause appreciable color changes within days. Products kept in dark or opaque packaging may be expected to retain color longer than those exposed to light.

Realizing the importance of creating quality reserve for the consumers, most large food industries in Europe are demanding much lower temperatures in the first links of the chain. Two main challenges for the years to come will be to improve our education for proper handling of frozen foods throughout the whole frozen chain and to find simple and inexpensive ways for control and checking

### V. CONCLUSION

Appropriate freezing and thawing processes and optimized temperature stability during storage preserve the quality and characteristics of fresh foods in the frozen product to a degree that is scarcely reached by any other preservation method. Since the glassy state is only readily attained by a very limited number of frozen food products, product deterioration in frozen storage is a continuous irreversible process. Physical and biochemical activities continue at  $-18^{\circ}$ C, but the reaction rates at this temperature have only a limited practical significance during the commercial life.

It must be borne in mind that raw material characteristics are the most important factors related to final frozen food quality. No process improves the intrinsic qualities of a

food product, but a most efficient way to preserve them over time always exists. The challenge of the freezing process is to reduce the rate of physicochemical deterioration through improved storage, particularly its temperature stability and packaging material and also through the quality of the raw material.

After the writing of this chapter a comprehensive review on the "Frozen Food Legislation" by L. B. Sørensen, particularly in the EU and the US, has been published in the *Bulletin of the International Institute of Refrigeration*, 2002-4 (8 pages, 11 ref).

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# 4

# **Frozen Food Packaging**

Kit L. Yam and Hua Zhao

Rutgers University, New Brunswick, New Jersey, U.S.A.

Christopher C. Lai

Pacteco Inc., Kalamazoo, Michigan, U.S.A.

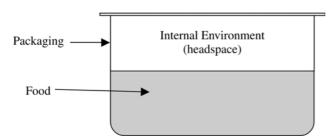
### I. FUNCTIONS OF PACKAGING

The basic functions of the package are to contain the food, protect the food, provide convenience, and convey product information. The package protects the food against physical, chemical, and biological damages. It also acts as a physical barrier to moisture, oxygen, volatile compounds, and microorganisms, which are detrimental to the food. The package provides the consumer with convenient features such as microwavability, resealability, single serving, and ease of use. The package conveys useful information such as product contents, nutritional values, and preparation instructions. All these functions are applicable to the packaging of frozen foods (1).

The food package can function best when integrated into a food packaging system, which involves certain physical components and operations. The major physical components are the food, the package, and the environment (Fig. 1). It is useful to divide the environment into internal and external—the internal environment (which may or may not include a headspace) is inside the package and is in direct contact with the food, and the external environment is outside the package and depends on the storage and distribution conditions. The operations are the manufacturing, distribution, and disposal of the food package. In designing the food packaging system, these physical components and operations must be considered to prevent overpackaging and underpackaging, which result in higher costs, lower quality and in some cases, health risks.

There are several requirements in the selection of packaging materials for frozen foods: temperature stability, barrier properties, thermal insulation properties, consumer appeal, and machine compatibility (2). Temperature stability is necessary since the packaging materials must be able to withstand the abuses encountered over a broad range of temperatures, including freezer temperatures during transportation and storage as well as high temperatures during the heating of the food package in the microwave or conventional oven. Barrier properties are necessary to minimize deteriorative effects of moisture, oxygen, and light to the food product. Thermal insulation helps maintain low temperatures for frozen foods during distribution and helps minimize temperature fluctuations that may cause degradation of the products. Consumer appeal is necessary for successful marketing of the products; the packaging materials should allow high quality

### External Environment



**Figure 1** Physical components of a packaging system.

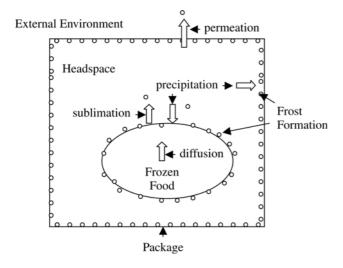
printing and graphics. Machine compatibility is necessary to ensure that the packaging materials are compatible with low-cost high-speed machineries.

Frozen food packages are typically made using carton machines, form-fill-seal machines, and pouch-forming machines. Methods of construction and operation of these machines may be obtained from the manufacturers and the literature (3).

### II. DETERIORATION MODES OF FROZEN FOODS

In addition to mechanical damages, frozen food products can also fail via several deterioration modes. The most important deterioration modes of frozen foods are related to the transport of moisture. The water molecules in ice exert a vapor pressure that increases with temperature. Water molecules tend to move from high concentration to low concentration.

Figure 2 illustrates the various transport mechanisms of water molecules (small circles in the figure) in a frozen food package. Diffusion of water can occur within the food if a concentration gradient exists (in most cases, in the direction from center to surface). Sublimation, evaporation of water from ice to vapor, can occur at the food surface.



**Figure 2** Transport mechanisms of water molecules.

Precipitation of water vapor as ice crystals can occur on the food surface or on the interior package surface. Permeation is the transport of water vapor across the package walls, and the water vapor transmission rate (WVTR) is determined by the permeability of the package. The transport of water molecules can result in dehydration and frost formation, two of the major deterioration modes of frozen foods. In addition to water molecules, the transports of oxygen, flavor, and odor compounds are also important to frozen foods.

Dehydration in frozen foods, also known as freezer burn or desiccation, is the moisture loss at the product surface due to sublimation of ice. The moisture loss results in a drier product surface and a concentration gradient that can cause water molecules to diffuse from the food center to its surface. Dehydration is a major deterioration mode in frozen food since it reduces product weight and adversely changes product appearance, texture, and taste. For example, when proteins in meat, poultry, and fish products become irreversibly dehydrated, the tissues become dry and tough. These products frequently contain considerable amounts of fats and oils, and dehydration can make these fats and oils more susceptible to oxidation by opening up the tissues and thus making more surface areas available for oxidation.

If the food product is unprotected (i.e., without package), the rate of moisture loss to the external environment is rapid. To retard moisture loss, protecting the product by a good moisture barrier package is necessary. In addition, the package should also have good tensile, tear, and burst strength at low temperatures; otherwise, package damages (such as holes or cuts) can occur and cripple the protective function of the package.

Frost formation is a phenomenon by which water vapor precipitates as frost on the food surface or on the interior surface of the package. Frost formation contributes to the problem of freezer burn, since moisture is removed from the product, and it also makes the package less appealing to the consumer. A major factor that affects frost formation is headspace volume: in the presence of headspace, moisture loss occurs from the food surface to the headspace through sublimation, even when the food is protected by a good moisture barrier package. It is the water vapor in the headspace that is responsible for frost formation. Therefore an effective packaging technique is to wrap the food product tightly to eliminate the headspace and its water. Another major factor that affects frost formation is temperature fluctuation. Since vapor pressure is temperature dependent, any temperature fluctuations can result in different vapor pressures at different locations, and thus a concentration gradient is created that tends to accelerate the rates of sublimation and precipitation.

Oxidation is another deterioration mode for frozen foods. Although oxidation occurs slowly at freezer temperatures, it remains a problem, since frozen foods are often stored for prolonged periods of time and oxygen is more soluble in food at lower temperatures. Oxidative reactions can result in rancidity, off-flavor, and pigment discoloration in frozen meat and seafood products. In general, oxidation reactions accelerate with increasing amounts of oxygen present, but there are exceptions. Different foods have different susceptibilities to oxidation; for example, pork and poultry are more susceptible than beef and veal to oxygen. To protect oxygen sensitive frozen foods, the package should have low oxygen permeability.

Flavor loss is also a deterioration mode for frozen foods. Some flavor compounds are volatile and exert considerable vapor pressures even at freezer temperatures. The alternation of flavor profile due to flavor loss may cause the consumer to reject the product. Odor pickup is also a deterioration mode. Trimethylamine, a compound responsible for the objectionable "fishy" flavor, is volatile at temperatures as low as

-23°C. Therefore the package should also have low permeability to flavor and odor compounds.

It is clear from the above discussion that packaging is vital for protecting frozen foods. Understanding the deterioration modes can help to develop packaging strategies to extend the shelf lives of the products.

### III. PACKAGING MATERIALS

Packaging materials include paper, plastics, glass, and metal. For packaging frozen foods, paper and plastics are most commonly used, metal is occasionally used (for example, as metal ends in composite cans for frozen concentrated juice), and glass seldom. In some package designs, combinations of paper and plastics are used: for example, a frozen meal may be placed inside a plastic tray with a lid, and the tray is placed inside a paperboard carton. The major roles of the package are to protect the products against mechanical damages and deteriorative effects of gas and vapor at low temperatures.

# A. Paper and Paperboard

Paper and paperboard are mainly used to provide structural support and protect the frozen food products from mechanical damages. These materials are sometimes used as a light barrier, but their moisture and oxygen barrier properties are poor. These materials are made of wood fibers containing cellulose, hemicellulose, and polymeric residues. They have the advantages of good structural strength, low cost, recyclability, and good printability.

There are several types of paper used for frozen food packaging. Kraft paper is a coarse paper, which may be used in unbleached or bleached form. Greaseproof paper and glassine paper provide good protection against oil and grease. Waxed paper is a good moisture barrier and can serve as a heat-sealable layer. Paper is sometimes used as an insert to separate individual items (such as beef patties) within the same package so that those items do not stick together on freezing.

Paperboard is commonly used for individual packages and secondary packages (e.g., a box that contains several individual packages). Waxed cartonboard, with a moisture-proof regenerated cellulose film overwrap, was used as earlier packaging for frozen vegetables and fruits (4). There are two basic folding paperboard designs: skillet and three-flap closed carton (3). Bleached Kraft carton is often used in packaging for frozen foods owing to its strength and good appearance. To improve moisture and oxygen barrier qualities, paperboard is sometimes coated or laminated with plastics or aluminum. To improve its appearance and printing quality, the paperboard is sometimes coated with clay and other minerals. It is quite common that a plastic bag containing a frozen food product is placed inside a paperboard carton. In this case, the plastic bag provides the gas and vapor protection, and the carton provides the structural support and mechanical protection.

### B. Plastics

Many frozen food products are packaged in plastics for moisture, oxygen, flavor, and odor protection. Plastics consist mainly of synthetic polymers and small amounts of additives (e.g., antioxidant and pigment) which can be cast, extruded, and molded into various

shapes such as films, sheets, and containers. Most polymers used in food-packaging plastics have molecular weights between 50,000 and 150,000. Plastics provide a wide range of properties relating to mechanical strength, gas barrier capability, printability, heat performance, and machine performance (5).

Plastics have a wide range of gas and vapor barrier properties offering many choices for different package requirements. The gas barrier properties of a plastic packaging material are usually quantified in terms of permeability: the lower the permeability, the better the gas barrier. Permeability is a function of the plastic material, permeant gas, temperature, and in some cases relative humidity.

In selecting plastic materials for frozen foods, it is necessary to select those which remain flexible at freezer temperatures. Abuse testing (usually includes a combination of shipping, vibration, compression, and drop tests) should be conducted to ensure that the package is not brittle and that it does not lose its product integrity at low temperatures.

The following is a general discussion of the basic food packaging polymers (Fig. 3). These polymers are mostly used as bags or pouches for packaging frozen foods. Relatively thick films are used to protect against frozen food products (such as crab legs) which have cutting edges or sharp points. Some packaging films are coextruded or laminated multilayer films consisting of several layers of different polymers. By wisely selecting different polymers, multilayer films can offer the advantages of lower cost and/or better performance.

# 1. Polyethylene (PE)

PE is a commonly used polymer for plastic bags for individually quick frozen (IQF) foods (e.g., vegetables, fruits, shellfish) (2). The advantages of PE are low cost, easy processing,

Ethylene-vinyl acetate (EVA) copolymer

**Figure 3** Chemical structures of some common food packaging polymers.

and good mechanical and printing properties. PE is usually classified into high-density polyethylene (HDPE), low-density polyethylene (LDPE), and linear low-density polyethylene (LLDPE). These classifications differ in density, chain branching, and crystallinity.

HDPE is a linear polymer with relatively few side chains. Its density is typically between 0.94 and 0.97 g/cm<sup>3</sup>. It has a higher melting point than LDPE (135°C versus 110°C typically) and thus is more suitable for high-temperature application such as boilin-bag applications (2). HDPE is used in films and containers for frozen foods.

LDPE normally has a density range of 0.91 to 0.93 g/cm<sup>3</sup>. It is a branched polymer with many long side chains. LDPE is used mostly as film, as an adhesive in multilayer structures, or as waterproof and greaseproof coatings for paperboard packaging materials. The film made from LDPE has the advantages of low cost, softness, flexibility, stretchiness, clarity, and heat sealability.

LLDPE is a copolymer with many short side chains. It has LDPE's clarity and heat sealability, as well as HDPE's strength and toughness. Therefore, LLDPE has substituted for LDPE in many food-packaging applications.

## 2. Polypropylene (PP)

PP has the lowest density ( $\sim 0.9$ ) among all major plastics. It has higher tensile strength, stiffness, and hardness than PE. PP cast film is clearer than PE film and is used in applications where transparency is required.

# 3. Polyvinyl Chloride (PVC)

PVC is a clear, hard polymer which is often modified with plasticizers (organic liquids of low volatility). Plasticized PVC films are limp, tacky, and stretchable, and the films are commonly used for packaging meat. PVC has better clarity, oil resistance, and barrier properties than those of HDPE.

# 4. Polystyrene (PS)

PS is a clear, hard, and low impact resistance polymer. High-impact polystyrene (HIPS) is formed by modifying PS with elastomeric molecules such as butadiene. HIPS is more suitable for freezer temperature applications because it has significantly higher impact resistance. Expanded PS (EPS) of various bulk densities are manufactured by adding foaming agents in the extrusion process. Some frozen seafood products (such as lobster tails) are vacuum-skinned down on an EPS tray with a coextruded film.

### 5. Polyethylene Terephthalate (PET)

PET is the major polyester used in food packaging that can tolerate freezer temperatures and high temperatures. It also provides good resistance to grease and moisture. The biaxial orientation of PET film can improve its clarity and mechanical properties. The crystallized polyethylene terephthalate (CPET) can withstand temperatures up to 220°C, and CPET food trays are suitable for use in microwave and conventional dual ovens.

### 6. Ethylene-Vinyl Acetate (EVA)

EVA is a copolymer containing 2 to 18% vinyl acetate. It has long chains of ethylene hydrocarbons with acetate groups randomly throughout the chains. EVA film is tough and

tacky, and thus it is often blended with polyethylene to improve sealability, stress resistance, and flex cracking resistance. EVA can be used as bags for frozen foods, and is coextruded with Surlyn ionomer and LDPE for the application in skin packaging.

# C. Barrier Properties of Plastics

For foods that are sensitive to moisture or oxygen, gas barrier protection is the major function of the package in providing adequate shelf life, the time period during which the food maintains acceptable quality. Controlling moisture loss is important for frozen foods because moisture loss (sublimation of ice) results in freezer burn and discoloration of the product. Oxidative reaction is also important for some foods even at freezer temperatures.

The transport of gases between the external environment and the headspace through the package can occur by means of leakage and/or permeation. For a properly sealed package in which leakage is not a problem, permeation is the major mechanism of gaseous transport.

Gas permeation is an important consideration in packaging foods with plastics, since food packaging plastics are permeable to moisture, oxygen, carbon dioxide, nitrogen, and other gases (including those that can cause off-odor problems). The gas permeation rate of most interest for frozen foods is the water vapor transmission rate (WVTR).

WVTR may be defined as the amount of water vapor transmitted through the package per day [g  $H_2O/(day(package))$ ] under specific conditions (usually 38°C, 90% relative humidity). WVTR can also be expressed more generally in terms of the amount of water transmitted through  $100\,\text{in}^2$  package area per day [g  $H_2O/(day(100\,\text{in}^2))$ ] where the package area is normalized.

If WVTR is assumed a constant, the shelf life  $(t_s)$  can be estimated by

$$t_{\rm s} = \frac{\rm H_2O_{\rm max}}{\rm WVTR} \tag{1}$$

where  $H_2O_{max}$  is the maximum allowable water (g  $H_2O$ ) which can be determined by sensory evaluation. In practice WVTR is not a constant but decreases with time, because headspace relative humidity decreases and concentration gradient decreases with time. Thus the actual shelf life is slightly higher than that predicted by Eq. (1).

For products that are oxygen sensitive, the oxygen transmission rate (OTR) of a plastic package from the external environment to the headspace can be expressed by

$$OTR = \frac{\overline{P}A}{L}(P_e - P_i)$$
 (2)

where OTR is oxygen transmission rate, cc  $O_2$ /day;  $\overline{P}$  is oxygen permeability (cc  $O_2$  (mil)/ (100 in² (day) (atm)); A is surface area of the package, in²; L is thickness of the package, mil;  $P_e$  is oxygen partial pressure in the external environment, atm; and  $P_i$  is oxygen partial pressure in headspace, atm.

The shelf life  $(t_s)$  can be calculated using

$$t_{\rm s} = \frac{{\rm O}_{2,\rm max}}{{\rm OTR}} \tag{3}$$

where  $t_s$  is shelf life, day, and  $O_{2,max}$  is maximum allowable oxygen, cc  $O_2$ . It has been assumed in Eqs. (1) and (3) that permeation through the package (not the rate of deterioration) is the major factor limiting the shelf life. This is a reasonable assumption

when packaging materials of low permeability are used. Combining Eq. (2) with Eq. (3) gives

$$t_{\rm s} = \frac{{\rm O}_{2,\rm max}L}{\overline{P}A(P_{\rm e} - P_{\rm i})} \tag{4}$$

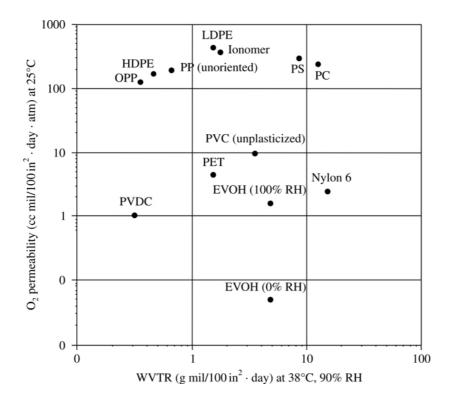
Equations (1) through (4) can be used to evaluate many what-if scenarios. For example, if the thickness of the package is decreased by 20% and the surface area is increased by 20%, then the above equation predicts that the shelf life will be decreased by 33.3%.

An obstacle is that permeability values of packaging polymers are generally not available at freezer temperatures. It is mostly due to the time and cost necessary for measuring permeability at low temperatures. However, literature permeability values are available at higher temperatures, and Fig. 4 shows the relationship between oxygen permeability and WVTR for some commonly used food packaging plastics (5). In practice, one can use literature data as a reference when comparing different polymers; better still, one can measure the permeability of interest.

# IV. PACKAGING TECHNOLOGIES

# A. Vacuum Packaging and Modified Atmosphere Packaging

As mentioned earlier, the headspace is an important factor that affects several deterioration modes of frozen foods. The water vapor in the headspace can cause frost formation, and the oxygen in the headspace can cause oxidation. A technique to control



**Figure 4** Gas barrier properties of common food packaging polymers.

the headspace is vacuum packaging, which simply involves removing air from the headspace. This technique has been shown to help maintain the quality of various frozen products including pizza, seafood, beef and pork (6). There are two forms of vacuum packaging, depending on the rigidity of the package.

The first form of vacuum packaging involves a rigid package (e.g., glass jar) or a semirigid package (e.g., plastic container) in which most of the air is evacuated, but a headspace still remains in the package. The removal of air typically reduces the oxygen level in the headspace to as low as 1%, which significantly helps to reduce the problem of oxidation. However, frost formation and freezer burn are still problematic since the headspace exists.

The second form of vacuum packaging involves a flexible package (e.g., a plastic pouch) in which not only the oxygen is removed but also the headspace is eliminated. Thus both oxidation and frost formation are controlled. This form is also known as vacuum skin packaging, since the food is tightly wrapped by the package. The mechanical stress created by the vacuum also helps to remove air pockets inside the product. This technique has been widely used to package frozen meat and seafood products including meat balls, clam strips, lobster tails, salmon, and farmed rainbow trout (7). Several types of materials are used for vacuum skin packaging, such as a blend of Surlyn ionomer resin with low-density polyethylene (LDPE) and ethylene-vinyl acetate (EVA) (8).

Modified atmosphere packaging (MAP) is a technique that involves replacing air (especially its oxygen) in the headspace by other gases such as nitrogen and carbon dioxide. Nitrogen is used as an inert gas filler, and carbon dioxide is used because of its ability to inhibit microbial growth. MAP is seldom used for frozen foods, because vacuum packaging is often a better alternative in terms of cost (no gas required) and effectiveness (no frost formation). However, MAP is used in some refrigerated and shelf-stable food products where the benefit of carbon dioxide is justified or the products cannot withstand the mechanical stress of vacuum packaging.

Both vacuum packaging and MAP require the use of gas barrier packaging materials; otherwise, the vacuum or the modified atmosphere cannot be maintained for a prolonged period of time. Vacuum equipment or gas flushing equipment is also required.

### B. Time-Temperature Indicator (TTI)

It is critical to maintain frozen food products at constant low temperatures. Temperature abuses due to improper handling may result in lower food quality and even worse, microbial growth if the abuse is severe. Monitoring temperature is a critical control point for frozen foods in designing a HACCP (hazard analysis critical control points) program. While temperature recorders (such as handheld electronic temperature monitoring devices) are often placed in storage rooms and trucks to monitor temperatures, these recorders are not attached directly to the food packages.

A time-temperature indicator (TTI) is a small self-adhesive label that can be attached to a food package for monitoring the package temperatures from the time of production to the time of consumption. The TTI helps to determine whether the product is still fresh at the point of purchase and at home by providing the consumer with a visual indication.

An important aspect of the TTI is the visual indication system, which typically involves color change or size change associated with diffusion, chemical reaction, or enzymatic reaction. The visual indication (color change or size change) is correlated to the temperatures or the time–temperature history. In order to use the TTI to indicate the shelf

life of the product, the kinetics of the TTI and the kinetics of the food must be known, and it is also necessary to match the activation energies of the TTI and the food deterioration reaction. The technical details are beyond the scope this chapter but they can be found elsewhere (9).

There are two common types of TTIs. The first type is the temperature limit indicator (or threshold indicator), which triggers an indication when a certain temperature limit is exceeded. For example, if the upper limit is set at  $-3^{\circ}$ C, the TTI will trigger a color indication once the temperature limit is exceeded. The second type is the time-temperature integrator, which triggers an indication when the time-temperature limit is exceeded. For example, if the indicator is set at 60 days and  $-18^{\circ}$ C, the TTI will trigger a color indication once an equivalent of this time-temperature history is exceeded. The equivalent time-temperature history is estimated from the kinetics of the TTI.

Presently several TTIs are available in the market. The LifeLines Fresh-Check<sup>®</sup> is based on a polymerization reaction which responds to cumulative exposure to temperature. The 3M Monitor Mark<sup>®</sup> is based on dye diffusion, which is activated by pulling out an activation strip. Upon exposure to temperatures above the threshold, the activated indicator's window irreversibly turns blue, warning that product quality testing should be performed. The Vitsab<sup>®</sup> TTI is based on enzymatic color change. More information about these TTIs can be found on the company websites.

### V. CONCLUDING REMARKS

Packaging is essential for protecting frozen foods from mechanical damage, moisture loss, flavor loss, odor pickup, and oxidation. Reducing headspace and air pockets inside of the package by vacuum packaging will help minimize frost formation.

One needs to analyze both the distribution environment and the stability of the product in order to formulate the packaging requirements for the specific product. In analysis of food stability, the dominant deterioration modes should be identified. Their kinetics and acceptable limits should also be determined. Once the food stability and required shelf life is known, package requirements for protecting the product from the dominant deterioration modes can be decided.

It is helpful to work with the packaging material supplier and packaging machine manufacturer to design and manufacture the package according to the requirements. Other factors including those described earlier in this chapter should also be considered in the package design process.

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### 5

# Frozen Food Components and Chemical Reactions

Miang H. Lim, Janet E. McFetridge, and Jens Liesebach University of Otago, Dunedin, New Zealand

#### I. INTRODUCTION

One of the greatest challenges for food technologists is to maintain the quality of food products for an extended period. A decrease in temperature generally decreases the rate of chemical reactions that are responsible for the deterioration in food quality over time, therefore freezing is frequently used to extend the shelf life of food products. However, freezing is not a perfect method of preservation, because even at low temperatures deterioration of quality may still occur. The formation of ice may result in textural changes and disruption of cell compartments that cause the release of chemically reactive components. Furthermore, the removal of water during ice formation concentrates the solutes in an unfrozen matrix, which can affect reaction conditions, such as pH and ionic strength. Therefore in order to extend the shelf life of frozen food products, it is crucial to understand the chemical reactions that can occur in food components that can lead to quality deterioration.

The effect of freezing on the food components is diverse, and some components are affected more than others. For example, protein can be irreversibly denatured by freezing, whereas carbohydrates are generally more stable. This chapter focuses on chemical and biochemical reactions that affect the quality of frozen food systems. These reactions and specific examples in food are summarized in Table 1. Methods for reducing the rate of deterioration are also discussed in this chapter.

#### II. CHEMICAL AND BIOCHEMICAL REACTIONS IN FROZEN FOOD

#### A. Protein

Protein may undergo changes during freezing and frozen storage, primarily because of denaturation. Denaturation can be defined as a loss of functionality caused by changes in the protein structure due to the disruption of chemical bonds and by secondary interactions with other constituents (1). Alterations of the secondary and higher structures of proteins affect the spatial arrangement of the polypeptide chain with respect to other neighboring polypeptide chains (1, 2). Structural and spatial alterations can cause a range of textural and functional changes, such as the development of toughness, loss of protein

 Table 1
 Chemical Reactions of Food Components that Affect Food Quality

Food components	Mechanism of degradation	Effect on food quality	Studies in food
Protein	Denaturation	Degradation of texture and functional properties	Toughening and functional changes, particularly loss of protein solubility, in fish (4, 12, 14, 19) Loss of protein solubility, emulsifying capacity, and water holding capacity of meat for processing (3)
Lipid	Hydrolysis	Release of FFA—known to contribute to unpleasant flavors	Short chain FFA giving rancid odor in dairy product (85)
		·	Medium chain FFA caused a "sweaty" flavor in mutton (86)
			Toughening of muscles in frozen Indian oil sardine (8)
	Oxidation	Unstable hydroperoxides break	Off-flavors shelled oysters (35)
	Autoxidation Enzymatic oxidation	down to reactive compounds and interact with other components to produce off- flavors, discoloration, and toughening of muscle protein	Detection of rancid flavor in silver pomfret (87)
Carbohydrates	Hydrolysis	Increases the amount of smaller molecular weight components—leads to lower melting temperatures	Sucrose hydrolysis (46)
		Change of texture	Firmness of ice cream decreased as hydrolysis progressed (45)
Color pigments			
(a) Chlorophyll	Pheophytinization	Green chlorophyll forms olive- brown pheophytin in the presence of acid or heat	Greenness in Brussels sprouts decreased (77) Stability of green color in kiwifruit (53, 54) Frozen blanched spinach had a higher amount of pheophytin than fresh (88)

(b) Anthocyanin	Enzymatic reaction	Glucosidase hydrolyses glycosidic linkages and produces sugars and aglycone compounds	Loss of anthocyanin in raspberry in the late cultivar was more severe than the early cultivars (57)
	Structure of anthocyanin depends on pH value	Depending on the pH of the food, different forms of anthocyanin exist, usually from red to blue as pH increases	Red color hue of sour cherry weakened during
(c) Carotenoids	Oxidation	The loss of pigments causes fading of color and loss of nutritive value	Loss of carotenoid in salmon (40)
Flavor compounds	Enzymatic degradation	Change of flavor profile	Change in the composition of aromatic compounds of strawberries (64–66)
			Change in aroma profile of frozen green peas (67)
	Lipid oxidation	Produces off-flavors	Green and fatty off-flavor notes in frozen trout due to breakdown products of unsaturated FFA (71)
	Leaching of components during the blanching process	Weakens and changes the sensory perception	Decrease of organic acids in green beans and Padron peppers (69)
			Change in volatile composition of guava (73)
	Effect of heat		Changes in concentration of odorants during heat treatment (66)
Micronutrients			· /
(a) Vitamins	Oxidation	Loss of nutritional value because of the loss of vitamins	Loss of vitamins C and B6 due to blanching in french fries (78)
			Oxidation of AA in peas, lima beans, corn and green beans (79)
(b) Minerals	Generally stable		Unchanged mineral composition in artichokes, green beans, and peas after freezing (84)
	Loss mainly through leaching		Mineral content of boiled fresh vegetables was not different from frozen vegetables (75)

solubility, loss of emulsifying capacity, and loss of water holding capacity (3–5). The myofibrillar proteins (mainly myosin and actomyosin) are considered to be the most susceptible to denaturation, while sarcoplasmic proteins have been reported to be more stable (6). For example, in freshwater whitefish stored for 16 weeks at  $-10^{\circ}$ C, the actomyosin solubility decreased by 50% but the sarcoplasmic proteins were unaffected (4).

In addition to the physical and chemical changes associated with ice formation, loss of protein stability may also be caused by interactions of protein with other components and by enzyme activity. These factors have been reviewed by several authors as causes for denaturation in fish proteins (1,6,7). Lipid oxidation has been shown to be highly correlated with protein denaturation during freezing. For example, the oxidation and hydrolysis of lipids in sardines at  $-20^{\circ}$ C was inversely related to protein solubility and other functional changes (8). The addition of malonaldehyde, a commonly occurring product of lipid oxidation, to trout myosin solutions during storage at -4°C accelerated protein denaturation (9). On the other hand, actomyosin in fatty fish, such as halibut and rosefish, is more stable than the actomyosin in lean fish, such as cod and haddock, after one year of frozen storage at  $-23^{\circ}$ C (10). The actomyosin from fatty fish had little or no denaturation, whereas that from lean fish was almost completely denatured. Enzymes, particularly trimethylamine oxide demethylase (TMAO demethylase), have been linked to protein denaturation. TMAO demethylase hydrolyzes trimethylamine oxide (TMAO), which is naturally present in some fish and shellfish, into dimethylamine (DMA) and formaldehyde (7). Formaldehyde is reported to form covalent cross-links with protein and therefore accelerating denaturation (11). Myofibrillar patterns of hake muscle soaked in formaldehyde had a higher amount of high molecular weight aggregates caused by covalent cross-linking of structural proteins (12). The effect of the addition of formaldehyde on actomyosin denaturation during frozen storage has also been studied in hake, pork, and chicken (13). The stability of the actomyosin in the presence of formaldehyde differed according to species, with chicken being the most stable to formaldehyde.

Cross-linking of polypeptide chains due to disulfide bond formation may also contribute to protein denaturation in frozen fish, although it may be a secondary factor (14–16). In minced halibut stored at  $-10^{\circ}$ C, insolubilization of the myofibrillar proteins was reduced when a disulfide bond reducing agent, such as  $\beta$ -mercaptoethanol, was added (14). Disulfide bond formation has also been implicated as a factor contributing to the loss of water holding capacity in haddock during frozen storage (12).

Various processing factors may influence protein denaturation during frozen storage. The factors include the handling and processing prior to freezing, the temperature of storage, and the rate of freezing. Mincing has been shown to promote toughening in frozen fish, for example minced hake accumulated formaldehyde at a faster rate than fillets (11). Depending on the species, aging fish on ice prior to freezing may be advantageous to quality (15). Aging red hake on ice before processing and frozen storage was shown to produce a more acceptable product than other treatments, such as the removal of dark muscle or dipping in sodium tripolyphosphate or erythorbate (17).

The kinetics of protein denaturation is influenced by temperature. At temperatures just below the freezing point, denaturation is very rapid. For example, the maximum rate of protein denaturation in rabbit and trout myosin solutions was reported to be at approximately  $-10^{\circ}$ C (9). Lowering the storage temperature generally reduces the degree of protein denaturation (12, 18, 19). In whiting fillets stored at  $-20^{\circ}$ C and lower, losses in protein extractability, calcium ATPase activity, and gel forming ability were reduced (19). However, there was no significant improvement in the quality of the whiting fillets when

stored below  $-20^{\circ}$ C. Similar results were observed in halibut (5). The differences in the development of toughness and off-flavors were insufficient to justify lowering the temperature below  $-20^{\circ}$ C.

Protein stability is also affected by the freezing rate. In general, faster freezing rates result in less damage, because smaller ice crystals are formed. However, fluctuating temperatures during frozen storage, due to overloading, power failures, and equipment breakdowns, can lead to the growth of ice crystals, which could counteract the benefit of using fast initial freezing rates (20). Storage temperature is therefore believed to be a more critical factor than freezing rate.

#### B. Lipids

Lipids can degrade in frozen systems by means of two well-known chemical processes: hydrolysis and oxidation (21). These processes lead to undesirable changes in the nutritional and sensory quality of foods, such as the production of rancid flavors and discoloration. The hydrolysis of lipids, also known as lipolysis, results in the release of free fatty acids, which can alter the flavor of frozen products. The rate of hydrolysis depends on storage temperature and time, and the type of food product (22–24). In frozen foods, lipid hydrolysis is generally catalyzed by enzymes. Both phospholipase A and lipases from muscle tissues are responsible for the hydrolysis of phospholipids and neutral lipids in frozen fish (24).

Lipid oxidation is a free radical reaction involving three major steps: initiation, propagation, and termination. Oxidation is frequently initiated by a spontaneous reaction with oxygen (autoxidation) or by an enzymatic reaction (25). Hydroperoxides are the initial products of lipid oxidation. These compounds are unstable and subsequently enter into numerous complex reactions that involve further degradation and interaction with other food components, causing off-flavors, discoloration, loss of nutrients, and toughening of protein (8, 21, 26). The rate of reaction increases with the degree of lipid unsaturation, therefore lipid oxidation is particularly a problem in high-fat fish (27).

Lipid degradation can be reduced in frozen foods by the lowering of storage temperatures, by the exclusion of oxygen, by adding antioxidants, and by supplementation of antioxidants in the diet of animals. The effect of lowering the storage temperature from  $-10^{\circ}$ C to  $-30^{\circ}$ C on lipid oxidation and hydrolysis was studied in cod and haddock (28). At  $-30^{\circ}$ C, lipid degradation in both species was reduced to the same extent, but at  $-10^{\circ}$ C the haddock was more susceptible to lipid degradation than the cod. Since oxygen is a key component in lipid oxidation, limiting its presence by modified atmosphere packaging can be beneficial to the shelf life of food, as shown in frozen pork (29), chicken and turkey (30, 31), shrimp (32), and pulverized niboshi (boiled and dried sardines) (33). The quality of processed fish fingers stored at  $-20^{\circ}$ C was shown to be influenced by the thickness of the packaging and the composition of the packaging material. The control, which was kept without packaging, was discarded after four weeks of storage due to rancid odors, a tough texture, and undesirable color changes. Fish fingers packed in 62.5 µm thick low-density polyethylene/high-density polyethylene (LD/HDPE) remained in a satisfactory condition for 28 weeks. The same product packaged with 100 µm thick low-density polyethylene/ Nylon-Primacore (LDPE/NY/PC) was stable for 32 weeks (34).

Adding antioxidants, such as butylated hydroxytoluene (BHT) and natural vitamin E, reduced lipid oxidation in shelled oysters during storage at  $-20^{\circ}$ C (35). Frozen rock oysters glazed with 1% sodium ascorbate alone or with 1% monosodium glutamate was efficient in extending their shelf life (36). Similar results were observed in frozen processed

turkey meat treated with antioxidants (a mixture of BHT, propyl gallate, and citric acid) where the degree of lipid oxidation was reduced (37). In addition, supplementing the diet of farm animals with vitamin E was shown to reduce lipid oxidation in meat (38, 39). Antioxidants may also occur naturally in food. The naturally occurring color pigment astaxanthin has been shown to reduce rancidity in farmed trout (40).

Other factors influencing lipid degradation include handling prior to freezing and the rate of freezing. Precooking of meat prior to freezing can increase lipid degradation during frozen storage, because the denatured heme proteins and ferrintin release free iron that catalyzes oxidation (41). It was shown that rancid flavors and thiobarbituric acid (TBA) values increased in precooked chicken meat during two to four months of frozen storage (42). Rapid freezing of chicken meat in liquid nitrogen produced a product with a lower degree of rancidity than air-blast freezing (42). Similar results have been observed in pork sausage (43).

#### C. Carbohydrates

Carbohydrates are susceptible to hydrolysis during frozen storage, as observed in frozen papaya (44). Sugar hydrolysis increases the number of moles of solutes in the food matrix, thus depressing the freezing temperature. This leads to a reduction in the amount of ice in the product, which may alter certain physical properties; for example, the firmness of ice cream was inversely related to the degree of hydrolysis (45). In a model system, the rate of sucrose hydrolysis, by enzymes and under acidic conditions, was investigated at low temperatures (46). The results indicated that even at temperatures as low as  $-22^{\circ}$ C, hydrolysis takes still place.

#### D. Color Pigments

The color of raw red meat tissues is mainly attributed to myoglobin. Carotenoid compounds, including astaxanthin, canthaxanthin, and  $\beta$ -carotene, contribute to the color of some marine products and poultry (47). Most of the color in fruit and vegetables is provided by chlorophylls (green leafy vegetables), carotenoids (orange, yellow, and red fruits and vegetables), and anthocyanins (the majority of berries and red to purplish vegetables).

The stability of color pigments during frozen storage is affected by treatment prior to processing and by processing and storage conditions (light, oxygen, heavy metals, temperature, water activity, pH, oxidizing, and reducing agents). Furthermore, oxidoreductase enzymes, such as lipoxygenase, catalase, peroxidase, polyphenol oxidase, and lipases, can cause bleaching of pigments and browning discoloration in fruits and vegetables (48, 49). Other chemical reactions within the food, particularly lipid oxidation, can also lead to changes in color.

Carotenoids are relatively stable during frozen storage. Sweet potato (50), tomato pulp (51), and green beans and broccoli (52) have shown minimal loss of  $\beta$ -carotene during the freezing process. The stability of meat colors, metmyoglobin in meat and carotenoids in fish and poultry, is generally decreased by lipid oxidation (26).

Chlorophyll and anthocyanins are considered to be less stable than carotenoids and can degrade through a variety of mechanisms. In the presence of heat and acid, chlorophyll changes from green to an olive-brown-colored pheophytin when the magnesium atom in chlorophyll is displaced by two hydrogen ions (47). Cultivar type and maturity have been shown to influence the stability of chlorophyll in kiwifruit during

frozen storage (53, 54). This may be because changes in total acidity and pH cause pheophytinization of chlorophyll (53, 54). However, there was no perceptible change in the greenness of parsley during storage at  $-20^{\circ}$ C for three years (55).

Anthocyanins are relatively unstable during frozen storage and are greatly affected by pH, organic acid content, sugar concentration, enzyme reactions, initial anthocyanin composition, and anthocyanin content. Anthocyanins are more stable under acidic conditions (47). In blackberries, maturity rather than cultivar was found to be more important for preventing blackberries from turning red during storage because the change in color was correlated with lower soluble solids, total anthocyanins, and higher titratible acidity (56). Glycosidases and polyphenolases are two classes of enzymes that promote loss of anthocyanin color intensity (47). Glycosidases hydrolyze cyanidin 3-glucoside, thus contributing to color loss in raspberries (57).

The choice of processing method can affect the stability of color pigments during frozen storage. Light and oxygen have been shown to accelerate color loss in tomato sauce (51), possibly by solubilization, isomerization, and degradation of lycopene (58). The lycopene content of tomato sauces prepared in an open kettle decreased faster during frozen storage than samples prepared in a tubular pasteurizer (58). The effect of still air freezing versus air-blast freezing on the color of pineapple slices was evaluated (59). The results showed that the freezing rate did not have any effect on the subsequent frozen storage stability of the pineapple color.

Various methods, such as blanching, modified storage atmosphere, and addition of antioxidants, have been used to enhance the stability of the color pigments in frozen foods. The main purpose of blanching prior to freezing is to inactivate enzymes that degrade color pigments. For example, lipoxygenase is known to degrade  $\beta$ -carotene and lutein (52). However, blanching can also lead to color losses. Heating can cause the development of an undesirable olive-green color in green vegetables because of chlorophyll degradation (52). Therefore minimization of blanching time is generally recommended (48). Modified atmospheres can be used to reduce color changes. For example, boiled shrimp flushed with nitrogen and sealed in a bag with low oxygen transmission reduced color fading and improved the overall quality of the shrimp (32). Furthermore, flushing precooked crabmeat with carbon dioxide was shown to prevent blue discoloration (60).

Color can also be stabilized using chemical additives such as antioxidants, applied either as a dip or added directly to the food product or even to the diet of the animal. Natural antioxidants, including ground tomato seeds and spices (rosemary and marjoram) have proved successful for color retention when added to tomato sauce to stabilize carotenoid pigments during frozen storage (51). Frozen avocado purees can be prevented from browning by adding sodium bisulfite and ascorbate prior to freezing (26). Browning in frozen crabmeat was reduced by a citric acid dip, but no significant reduction in the browning was observed with dips of sodium nitrite, ascorbic acid, sodium acid pyrophosphate, or brine with sodium citrate or tripolyphosphate (60). Sulfite can inhibit melanosis (blackspot) on crustaceans (61). However, if it is used in excess (2% for 10 minutes), it can cause decomposition of TMAO, thus initiating a chain of lipid and protein degradation reactions (61).

Dietary vitamin E supplementation for Holstein steers increased the color stability of the meat during storage at  $-20^{\circ}$ C (62). The color and carotenoid content of salmon fed with diets supplemented with astaxanthin was stable during frozen storage for up to 12 weeks at  $-20^{\circ}$ C, but salmon fed with cathaxanthin had significant loss in color scores and carotenoid content (63). In contrast, smoking of the fish significantly reduced the

carotenoid content of astaxanthin-fed salmon, but did not cause any decrease in color in canthaxanthin-fed Atlantic salmon during frozen storage (63).

#### E. Flavor Compounds

Food flavors are composed of volatile aroma compounds and taste components, such as organic acids and sugars. The effect of freezing and frozen storage on flavor compounds in food is variable; flavor changes are affected mainly by enzymatic activities and lipid oxidation. Freezing and thawing processes have been shown to change and weaken the flavor profile of fresh strawberries (64–66) and peas (67), but minimal changes were observed in the aroma volatile composition of raspberries studied under similar conditions (57). The flavors of fruit and vegetables may also be modified by the decrease in organic acids as observed in frozen kiwifruits, pineapples, and blanched green beans (54, 68, 69). In seafood, the development of off-flavors has been associated with the enzymatic degradation of TMAO that produces trimethylamine and dimethylamine (70). Another type of off-flavor in fish is caused by autoxidation of long chain  $\omega$ -3-unsaturated fatty acids, which produce carbonyl compounds characterized by an "oxidized fish oil" aroma (70, 71).

Blanching of vegetables prior to freezing can be used to inactivate the enzymes that cause changes to the flavor. The flavors of carrot, cauliflower, and beans during frozen storage have been stabilized by blanching (72). However, complete inactivation of enzymes may not be necessary, as it was shown that 5% residual peroxidase activity did not affect the quality of these blanched vegetables during storage (72). In contrast, the flavors of unblanched leek, onion, and swede did not change appreciably during frozen storage and were preferred to blanched products. The disadvantages of blanching with regard to flavor preservation are the loss of flavor compounds through leaching (69) and creation of a different flavor profile through heating (73). Therefore it is important to identify and specifically target enzymes such as lipoxygenases, lipases, lyases, and proteases for adequate blanching, rather than to utilize excessive blanching regimes for peroxidase destruction, which is commonly used as an indicator enzyme for vegetables (48). Foods, such as white truffle, that are unsuitable for blanching are susceptible to deterioration. These, along with the loss of other volatile compounds, were responsible for the typical truffle flavor (74).

Pasteurization is another heat pretreatment frequently used for frozen foods. Changes in the volatile composition of a pasteurized guava puree product were investigated during four months of storage at  $-10^{\circ}$ C and  $-20^{\circ}$ C (73). Pasteurized guava puree had increased levels of aldehydes and hydrocarbons and decreased levels of esters compared to unpasteurized guava puree. There was only a slight decrease in the concentration of alcohols and hydrocarbons in pasteurized guava puree stored at  $-20^{\circ}$ C.

#### F. Vitamins

Freezing is considered as one of the best processing methods for preserving nutrients in food. In a comparison of different storage methods, the nutrient content of frozen beans, sweet corn, and peas was similar to that of fresh vegetables that had been cooked by boiling. Frozen vegetables were higher in vitamin C, riboflavin, and thiamin than canned vegetables (75). Processing foods prior to freezing can result in vitamin losses through oxidation and leaching, with heat being a secondary factor (76). Generally, vitamin C has been used as a nutritional and quality marker for monitoring the effects of manufacturing

processes on nutrient losses, so it has been the focus of many studies in food (77). Vitamin C usually refers to the sum of both ascorbic acid and dehydroascorbic acid, since both are active forms of the vitamin (76). Beta-carotene is also commonly used to monitor the effect of processing on nutrient losses.

Blanching of foods prior to freezing can contribute to vitamin loss. The loss of 47% of ascorbic acid and 10% of vitamin B6 in commercially processed french fries during freezing was attributed mainly to the blanching process; the decrease caused by other unit operations was insignificant (78). Steam blanching is generally better for maintaining vitamin activity than hot-water blanching. Large-size cut french fries had better vitamin retention than small-size french fries because leaching was reduced. The effect of alternate freezing and thawing of peas, lima beans, corn, and green beans increased the destruction of ascorbic acid. This may have been due to an increase in the concentration of ascorbic acid and oxidative catalysts in the fluid of the tissue matrix (79).

The use of blanching to preserve nutrients depends largely on the type of food. Red peppers and kiwifruit, usually frozen without blanching, showed no significant loss of ascorbic acid during freezing (80). In contrast, the ascorbic acid content of unblanched okra and beans decreased by about 50% during freezing, while after blanching there was no significant change in the ascorbic acid content. The maximum decrease in ascorbic acid content in this study was observed at  $-1^{\circ}$ C to  $-8^{\circ}$ C (80). Another study showed that even though blanching reduced the vitamin C content of beans by 28%, it reduced further losses during twelve months of frozen storage (81). Unblanched beans and peppers lost more than 97% of their vitamin C content within one month of freezing, strongly indicating the need for blanching of vegetables before frozen storage in order to preserve nutrients. Similar results were obtained for broccoli and green beans (82).

#### G. Minerals

Minerals in food matrices may be present in many different forms such as chemical compounds, molecular complexes, and even free ions. Minerals present in any form can dramatically affect the color, texture, flavor, and stability of foods (83). Minerals cannot be easily destroyed by exposure to heat, light, oxidizing agents, or extremes in pH, but they can be lost through leaching or by physical separation of components. Even so, there were no changes observed in six mineral elements (Ca, Cu, Mg, Mn, Ni, and Zn) between fresh and frozen artichokes, green beans, and peas (84). Furthermore, the mineral content of boiled fresh vegetables was shown to be similar to that of boiled frozen vegetables (75).

#### III. CRYOPRESERVATION OF FROZEN FOOD

In the previous section, different techniques were discussed for reducing the rate of chemical reactions in foods, such as lowering the temperature, blanching, and modified atmosphere packaging. In conjunction with these techniques, further reductions in the rate of these reactions can be achieved by cryopreservatives, which can be natural components of the food or added as prefreezing treatments. Cryopreservatives can be classified as cryostabilizers or cryoprotective additives. Cryostabilizers are added to increase the viscosity of the frozen system, while cryoprotective additives are used to change or stabilize the structure of components that are susceptible to undesirable changes caused by freezing. The variety of frozen products may require special "cocktails" of cryopreservatives to be developed for each individual product (90).

The underlying principle of cryostabilization is based on increasing the viscosity of the frozen system, which controls the diffusion of solutes and therefore the rate of quality deterioration (91,92). When food is frozen, ice is formed and the solutes are concentrated in an unfrozen matrix. At the temperature of maximum freeze-concentration, where no more ice is formed, the unfrozen matrix undergoes a transition from viscous rubbery to a highly viscous glassy state. This temperature is called the glass transition temperature (Tg') (93). The extremely high viscosity associated with the glassy state limits the mobility of solutes and therefore reduces the rate of diffusion-controlled reactions.

Various types of reactions respond differently to this change in viscosity. Simple diffusion-controlled reactions, which have low activation energies (8 to  $25 \,\mathrm{kJ/mol}$ ), are especially sensitive to changes in the viscosity (94). The rate of lipolysis, lipid oxidation, and diffusion of <sup>14</sup>C-fructose were studied in mackerel mince at temperatures between  $-5^{\circ}\mathrm{C}$  and  $-20^{\circ}\mathrm{C}$  (95). The results indicated that the diffusion of <sup>14</sup>C-fructose and the production of thiobarbituric acid-related substances (TBARS) in mackerel mince decreased dramatically when the temperature was just below the Tg' of mackerel. However, the rate of lipolysis and accumulation of peroxides were only moderately influenced when stored below the Tg' of mackerel. Other studies have shown that ice recrystallization is reduced at temperatures below the Tg' of the unfrozen matrix (92, 96).

The rate of diffusion-controlled reactions has been studied in frozen systems by adding maltodextrins with high Tg' values (Tg' ranged from  $-10^{\circ}$ C to  $-18^{\circ}$ C) and carboxymethylcellulose (CMC, Tg' of  $-12^{\circ}$ C) (97). It was shown that in frozen systems containing maltodextrins the diffusion-controlled reaction rates were reduced at temperatures below their corresponding Tg', but frozen systems containing CMC were not reduced. From these results it was concluded that adding compounds with a high Tg' does not necessarily reduce the rate of diffusion-controlled reactions.

Typical cryostabilisers are high MW compounds, such as starch and starch hydrolyzates, which elevate the Tg' of the product (91,92). In general, low molecular weight components have a lower Tg' than high molecular weight components (92). The glass transition temperature is also affected by structural variation, such as polymer branching or linking of monomers (92). For instance, the Tg' for various disaccharides with a MW of  $342 \, \text{g/mol}$  can differ by up to  $7 \, \text{K}$  (92). Table 2 summarizes the Tg' and dextrose equivalent (DE) values for starch hydrolyzates.

**Table 2** Tg' and DE Values for Sugars and Polysaccharides

Solute	Tg' (°C)	Dextrose equivalent (DE)
Glucose	-43	100
Maltose	-31	53
Star Dri 42F <sup>a</sup>	-27	42
Maltotriose	-24	35
Maltoheptose	-13	16
Star Dri 20 <sup>a</sup>	-13	22
Star Dri 5 <sup>a</sup>	-10	5

<sup>&</sup>lt;sup>a</sup> Star Dri 5–42 corresponds to the dextrose equivalent, that is, the higher the number, the smaller the average molecular weight.

Source: Ref. 98.

Cryoprotective additives are widely used to protect protein from denaturation (90, 99). The additives interact with the surrounding water of the protein, and create a preferential hydration of the protein, and thus protects functional groups in protein from dehydration (99, 100). The structure of cryoprotective compounds is diverse. Several small sugars, polyols, amino acids, nucleotides, salts, and even surfactants have been shown to stabilize protein at subambient temperature (90).

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# 6

### Flavor of Frozen Foods

#### **Edith Ponce-Alquicira**

Universidad Autónoma Metropolitana, Mexico City, Mexico

#### I. INTRODUCTION

Flavor is one of the main attributes that together with color and texture influence the overall acceptability of foods. Generally, flavor is the result of a complex combination of sensations perceived by the two chemical senses, taste and smell. Taste is perceived by the taste buds on the tongue and other parts of the mouth, and it is usually described as sweet, sour/acid, salt, bitter, astringent, metallic, hot, cooling, and umami. The sense of smell detects certain odorous molecules in the air above the food that stimulate the olfactory receptors at the top of the nasal cavity. These volatile substances are detected before we eat, and during eating, as they pass in the breath from the mouth, through the posterior nares into the nasal cavity. Therefore compounds contributing to flavor can be divided into aroma and taste or nonvolatile compounds (1).

Nonvolatile precursors include lipids, peptides, amino acids, reducing sugars, vitamins and nucleotides, among others. Interaction of these compounds and/or their breakdown products generates a large number of intermediates and volatiles that contribute to the flavor development during processing and storage (2). Mechanisms by which flavor compounds are formed are important to improve the flavor of the starting materials, during the processing to gain optimum flavor, and during storage to maintain the flavor quality. This chapter describes the development of flavor precursors during processing, as well as off-flavors derived during freezing storage.

#### II. NONVOLATILE CONSTITUENTS

Nonvolatile or taste compounds are water-soluble; these can cause salty, sour or acid, bitter, sweet, umami, and hot or cool sensations. Salty taste is caused by the presence of inorganic salts as sodium or potassium chloride, together with monosodium glutamate and monosodium aspartate. Sweetness is produced by sugars (glucose, fructose, ribose) and some L-amino acids (glycine, alanine, serine, threonine, lysine, cysteine, methionine, asparagine, glutamine, proline, and hydroxyproline). Bitter tastes are generally caused by hypoxanthine, peptides such as anserine and carnosine, and the L-amino acids histidine, arginine, lysine, methionine, valine, leucine, isoleucine, phenylalanine, tryptophan, tyrosine, asparagine, and glutamine. Sour and acid tastes are caused by organic acids

(lactic acid and acetic acid), amino acids (aspartic acid, glutamic acid, histidine, and asparagine), and acidic phosphates. Hot and cool sensations or trigeminal responses do not contribute to the flavor of unprocessed foods, but addition of spices like chili and pepper usually contributes to the flavor of processed products. Finally, the umami taste has a characteristic savory quality supplied by glutamic acid, monosodium glutamate (MSG), 5'-guanosine monophosphate (GMP), 5'-inosine monophosphate (IMP) and some other peptides recognized by their flavor-enhancing properties (1, 2).

It has been suggested that amino acids, peptides, proteins, and nucleotides are the most important for the taste of red meats, fish, poultry, and dairy products (3). Meat flavor compounds are present in flesh and do not require cooking for their generation; but cooking and further processing may affect their concentration. It has been reported that concentrations of reducing sugars decreased by up to 20% during heating, while concentrations of free amino acids increased according to the cooking temperature from 55° to 95°C (4). Therefore changes in sugars, amino acids, and nucleotides that occur during processing and storage will affect not only the taste but also the overall flavor, because many of these compounds are precursors for the aroma components (1).

#### **III. AROMA CONSTITUENTS**

Aroma compounds are largely formed during processing by several mechanisms; most raw foods have none of the aroma of their processed foods. For instance, the characteristic aroma of cooked meat is largely generated during processing; where heating gives rise to several reactions that result in the formation of a complex mixture of chemicals. Aldehydes, ketones, and sulfur compounds provide the meaty, toasted, roasted, fatty, fruity, and sulfurous desirable meat aroma (2).

In the same way, major flavor compounds of sweet corn include 2-acetyl-1-pyrroline and 2-acetyl-2-thiazoline, dimethylsulphide, 1-hydroxy-2-propanone, 2-hydroxy-3-butanone, 2,3-butanediol, pyridine, pyrazine, alkylpyrazines, and 2-acetylthiazole. However, concentrations of volatiles in canned corn are many times higher than those present in frozen and fresh sweet corn (5). Moreover, fermented foods as Manchego-type cheeses develop their characteristic flavor during ripening because of lipoliytic and proteolytic enzyme activity (6). Furthermore, several routes intervene for the characteristic flavor formation of white bread; first, enzymes that regulate the metabolism of the grain produce the flavor precursors, which later are transformed by yeast during dough fermentation. Finally, nonenzymically browning reactions that take place during baking are responsible for the roasty, malty, and caramel notes. If the dough is fermented for longer time, 3-methylbutanol and 2-phenylethanol are formed in high concentrations and are responsible for the "yeasty" flavor impression (7).

Aroma profiling is also affected by handling and storage. For instance, more than 20 compounds have been reported to be responsible for yogurt flavor (acetone, lactic and acetic acids, diacetyl, acetaldehyde, and ethanol); during storage the acetaldehyde content increases, while acetic acid and diacetyl decrease, although acetone is unaffected (8).

In the same way, pasteurized guava puree shows a noticeable increase in ethyl alcohol, *n*-hexanal, decanoic acid, dodecanoic acid, and ethyl acetate when stored at 0 and  $-10^{\circ}$ C; but when they are stored at  $-20^{\circ}$ C, slight decreases in alcohols and hydrocarbons were detected (9).

The study of aroma is a complex task; many aroma compounds have relatively high odor thresholds and make little contribution to the overall flavor; others may be present at

very low concentrations, but owing to their very low thresholds, they have an enormous effect on flavor. In recent years, developments in analytical instrumentation and methodology have allowed the identification of many aroma compounds for several foods. Techniques for aroma collection include solvent extraction, concentration by means of adsorption through a static or dynamic gas-purging headspace (DHA) (8, 10–12), selective extraction using supercritical fluid extraction method (SFE) (13), and solid-phase microextraction (SPME) (14). Separation and identification is usually performed by gas chromatography—mass spectrometry (GC-MS) or gas chromatography—olfatometry (GC-O), using several ionization tools such as electron impact (EI), fast atom bombardment (FAB), field desorption (FL), laser desorption (LD), and electrospray (ESI).

#### IV. CHEMICAL REACTIONS RESPONSIBLE FOR FLAVOR

Interaction of nonvolatile precursors and/or their breakdown products generates a large number of intermediates and volatiles that contribute to flavor. The Maillard reaction, lipid oxidation, degradation of thiamine, and proteolytic and oxidative enzyme activities are the main mechanisms involved in flavor generation.

#### A. Maillard Reaction

The Maillard reaction is also known as nonenzymatic browning and involves several pathways that result in many aromatic products and high-molecular-weight melanoidins; high temperature, low water activity, and longer storage favor this reaction. The flavors produced can be pleasant or unpleasant and are typically described as caramelized, bready, nutty, roasted, or meaty. Coffee, roasted meats, bakery goods, and toasted nuts base their flavor on the Maillard reaction (15).

The initial stage involves the condensation of a free amine group and a reducing sugar to form a glucosylamine. The glucosylamines from aldose condensation undergo Amadori rearrangement to yield compounds such as 1-amino-1-deoxy-2-ketoses; while the condensation of ketoses is usually followed by the Heynes rearrangement to form 2-amino-2-deoxy-aldolases (16). The intermediate stage comprises dehydratation, fission, cyclization, and Strecker degradation; giving rise to a widely spectrum of compounds that modify the color, taste, odor and other properties of foods. The final stage involves condensation into high-molecular-weight melanoidins.

Concentrations of Amadori and Heyns compounds vary according to the reaction conditions; at pH 4–7, they can deamidate to give carbonyls such as deoxyosones, which yield many secondary products. The Strecker reaction involves transamination between deoxyosones and amino acids to produce amino-ketones, aldehydes, and CO<sub>2</sub>. Besides, the amino-ketones can yield pyrazine derivatives that together with aldehydes are powerful aroma constituents (7, 15, 16). Therefore volatile products formed by fission and aldehydes from Strecker degradation are mainly involved in the production of flavors and off-flavors. Aroma compounds come from simple sugar dehydration and fragmentation (furans, pyrones, cyclopentenes, carbonyls, acids), amino acid degradation (aldehydes, sulfur compounds) or further reactions (pyrroles, pyridines, imidazoles, pyrazines, oxazoles, thiazoles) (17).

#### **B.** Lipid Oxidation

There are three classes of lipids in foods, triacilglycerols, phospholipids, and cholesterol; all of them contribute to the perception and development of flavor. Phospholipids are located in membranes of cells and subcellular organelles, while triacilglycerols predominate in lipid droplets and fat depots. The proportion of saturated, monounsaturated, and polyunsaturated fatty acids (PUFAs) varies within foods and process.

Free fatty acids and triglicerides are capable of being oxidized by autoxidation or by enzymes called lipoxygenases; but polyunsaturated fatty acids are especially susceptible to autoxidation. The initial step of autoxidation involves the production of free radicals R( from lipids RH by their interaction with oxygen in the presence of heat, light, high-energy radiation, metal ions, or metalloproteins. Particularly, the heme iron of myoglobin accelerates lipid oxidation in meat and meat products, during cooking, as myoglobin denatures and iron is liberated. The free radical R( reacts to form a lipid peroxy radical ROO( that can react further to give a hydroxyperoxide ROOH. The second step or propagation provides further free radicals in a self-propagating chain process that can be terminated by a combination of two free radicals. The enzyme lipoxygenase is widely distributed in plants and animal foods; it is very specific about the substrate and how the substrate is oxidized leading to hydroperoxides. Lipid hydroperoxides are very unstable and break down to an alkoxy free radical, which decomposes, leading to undesirable rancid flavors and odors, mainly aldehyde products, including *n*-alkanals, *trans*-2-alkenals, 4-hydroxy-trans-2-alkenals and malonaldehyde (18). The type and source of lipids and the presence of lipoxygenase and inhibitors also affect the rate of lipid oxidation. Major volatile compounds generated from the oxidation of arachidonic and eicosapentaenoic acids by 12-lipoxygenase are 1-octen-3-ol, 2-octenal, 2-nonenal, 2-nonadienal, 1,5octadien-3-ol, and 2,5-octadien-1-ol. However, the addition of lipoxygenase inhibitors, esculetin, and BHA reduced the formation of volatiles (19). Susceptibility to oxidation also depends on the molecular differences of isozymes present in foods. As an example, the usual corn genotype and three genotypes varying in sweetness were blanched or unblanched, and stored for up to 12 months at  $-20^{\circ}$ C. All genotypes contained an isozyme of 80 kDa/pI 4.5, and sweeter genotypes also contained a peroxidase of 13.8 kDa; but after 12 months of frozen storage, an additional isozyme appeared in some extracts, giving rise to differences in the flavor profile (20).

#### V. OFF-FLAVOR DEVELOPMENT DURING FREEZING STORAGE

During frozen storage and distribution, foods are exposed to a wide range of environmental conditions such as temperature variations, reduced water activity, oxygen, and light, which trigger several chemical and physical changes. Deterioration in texture, flavor, and color are the most serious problems, causing shelf-life reduction, particularly when poor freezing practices are used or when the quality of the starting material is low (21). Fluctuating temperatures may cause recrystallization leading to undesirable sandy texture in dairy products, and phase changes involving melting and solidifying of fats on lipid-containing foods. In addition to temperature, other environmental factors such as oxygen, water activity, and pH induce chemical and enzymatic harmful changes. Deterioration of flavor involves rancidity, bitterness, or undesirable fishy taste, owing to the formation of low-molecular-weight compounds from lipid oxidation or protein

denaturation. Enzymes such as lipoxygenase, if not denatured during the blanching process, can also influence food quality even at subfreezing temperatures (21, 22).

#### A. Changes in Flavor of Frozen Foods Associated to Cell Damage

Freezing has a negative effect on the textural properties on several fruits and vegetables such as mashed potato, broccoli, blueberries, and strawberries. Freezing and fluctuation in temperature will produce cell damage by ice recristalization and dehydration. This may be reflected in a more open structure of the frozen product, which permits a greater ingress of oxygen, thus increasing oxidation, vitamin loss, and flavor deterioration (21, 23).

The open structure of frozen foods increases the migration of fluids containing cell nutrients that can facilitate the loss of micronutrients, microbial contamination, and the developing of off-flavors during thawing (24). Frozen and freeze-dried blueberries lose several flavor compounds, including the typical blueberry aroma (1,8-cineole) (25). On the other hand, very low temperature can promote the production of off-flavors in frozen-thawed strawberries when stored at -40 and  $-80^{\circ}$ C; freezing causes disruption of cells and decreases the pH of the cytosol, thus facilitating the subsequent release of sulphide ion as  $H_2S$ , and therefore off-flavors formation (26).

#### B. Changes in Flavor of Frozen Foods Associated to Lipids

Lipids play a multifunctional role in flavor; they influence both the physical (mouth feel) and chemical flavor perception, as lipids act as carriers for lipophilic flavor molecules, including off-flavors. Reduction in the fat content will result in higher flavor loss during storage due to flavor volatility. On a molecular level, triglycerides lower the vapor pressure of lipidic flavor compounds, thus increasing their thresholds (27).

Lipids also bring their own inherent flavor and are precursors for flavor development by lipolysis and lipid oxidation (28); there is a high correlations between reactions involving lipids and the development of off-flavors during storage. Lipid oxidation is influenced by several factors such as temperature and the presence of oxygen in the immediate vicinity of foods; but water activity also plays an important role, because at very low values it is found that in frozen food lipid oxidation occurs at high rates (7, 22).

The presence of unsaturated fatty acids in foods is a prime reason for the development of rancidity and off-flavors during frozen storage as long as oxygen is available. The generation of free radicals during rancidity also leads to undesirable reactions such as loss of vitamins, alteration of color, and deterioration of proteins and texture, which modify the whole flavor perception. Warmed-over flavor (WOF) is a flavor defect that occurs in reheated foods, and it is of special concern for precooked frozen foods. This occurs in cooked products that are stored under refrigeration or freezing conditions, where lipid oxidation initiates during storage and warm temperature of the reheating process accelerates the oxidation process with the outcome of rancid flavors, resulting in WOF (24). Furthermore, NH<sub>3</sub> exposition due to refrigerant leakage is related to rancid flavors in foods with a considerable reduction in the frozen storage life; off-flavors in lamb were detected by consumers after 3 to 6 months storage at  $-20^{\circ}$ C (29).

It is well known that such lipid oxidation products as hexanal and TBA reactive substances (TBARS) are highly correlated to the development WOF during frozen storage. Attempts have been made to reduce lipid deterioration during frozen storage by the addition of vitamins, antioxidants, or phosphate. Incorporation of a commercial oleoresin rosemary (OR)-coated salt (0.5 or 1 g/kg), sodium tripolyphosphate (STPP,

 $3 \, \mathrm{g/kg}$ ), or TBHQ (0.2 g/kg) in meat batters decreased hexanal production by 95% (30). Furthermore, dietary vitamin E (DL-alpha-tocopheryl acetate) equivalent to  $5 \times$ ,  $10 \times$ , and  $25 \times$  the NRC recommendations levels for the diet improved the oxidative stability and functionality of turkey breast meat and produced the most typical and acceptable meat flavor with the fewest oxidized off-flavor notes in fresh and frozen turkey meat (31). Besides, frozen meat from birds fed with docosahexaenoic acid (DHA) was more acceptable and stable owing to a higher lipid and flavor stability during storage at  $-23^{\circ}$ C for up to 82 days (32).

Ascorbic acid is mainly recognized as an antioxidant, owing to its radical and  $O_2$  scavenging effect. At low concentrations (800 ppm), ascorbic acid may act as a prooxidant, especially in the presence of metal-catalyzed oxidation. Ascorbic acid is able to reduce  $Fe^{3+}$  to  $Fe^{2+}$ . Nevertheless, the reduced  $Fe^{2+}$  catalyzes the breakdown of hydroperoxides, ROOH (ROOH +  $Fe^{2+} \rightarrow RO^{\bullet} + OH + Fe^{3+}$ ), to free radicals at a higher rate than  $Fe^{3+}$  (ROOH +  $Fe^{3+} \rightarrow ROO^{\bullet} + Fe^{2+}$ ) (33). Nevertheless, it has been reported that phosphatidic acid and phosphatidylserine can reduce the lipid oxidation catalytic activity of nonheme iron and hemoproteins (34).

The addition of phosphates [disodium phosphate (Pi), tetrasodium pyrophosphate (PP), sodium tripolyphosphate (TPP), sodium tetrapolyphosphate (TTPP), and sodium hexametaphosphate (HMP)] or sodium ascorbate monophosphate (SAsMP) in 0.3 and 0.5% levels reduces the formation of rancid and soapy flavors in frozen vacuum-packaged cooked turkey, with the additional decrease in cooking losses and the increase in moisture retention after thawing (35, 36).

Packaging also plays an important role to control WOF; in particular, modified packaging has been used to improve the flavor and aroma of cooked beef, where samples stored under vacuum and N2/CO2 atmospheres were more meaty, less warmed-over, less cardboardy, and less oxidized, and had lower TBA, than those in air-containing packages (37).

#### C. Changes in Flavor of Frozen Foods Associated to Proteins

Flavor release and perception is caused by interactions of flavor components with protein molecules by physical adsorption via van der Waals interactions, and by chemical reactions via covalent or electrostatic linkages, including salt, amides, ester formation, and condensation of aldehydes with NH<sub>2</sub> and SH groups. The specific distribution of hydrophilic and hydrophobic regions in proteins determines their shape, functionality, and binding of volatile compounds, so alterations in the protein moiety can lead to distortion of the sensory profile (38). Protein denaturation during freezing storage is due to several factors including changes in moisture, interaction with lipids or products derived from lipid oxidation, and the activity of specific enzymes as lipoxygenases and TMA-oxidase (21).

First, formation of ice crystals is conducive to dehydration and increases in salt concentration that promote protein aggregation. Distribution of hydrophilic and hydrophobic binding depends on the dielectric constant, pH, and ionic strength of the media. Thus high salt concentrations will disrupt the existing equilibrium leading to denaturation. It has been observed that denatured proteins irreversibly bind sulfurcontaining flavors by interchanging protein sulfhydryl groups and disulfides, where disulfides containing allyl or furfuryl groups are more reactive than saturated alkyl disulfides (39).

Free fatty acids derived from lipid hydrolysis can attach themselves hydrophobically or hydrophilically to the protein, creating more hydrophobic regions, resulting in a decrease of protein solubility (21). Moreover, free radicals can abstract a hydrogen from SH-side groups forming protein-free radicals that interact with other proteins or lipids forming protein-protein or protein-lipid aggregates.

Additionally, secondary lipid oxidation products such as malonaldehyde, propanal, and hexanal can react covalently with side-chain groups of histidine, methionine, cysteine, and lysine residues; these interactions increase the hydrophobicity of proteins and may decrease the flavor binding ability (21). In particular, aldehydes are more stable than free radicals and can diffuse into the cellular media inducing protein modifications (21, 40). In particular, products derived from lipid oxidation denaturate proteins such as myoglobin, where the heme iron is exposed, increasing its prooxidant activity (41). However, it has also been reported that  $\alpha,\beta$ -unsaturated aldehydes enhance the oxidation of ferrous oxymyoglobin to ferric metmyoglobin more than their saturated counterparts (40, 42).

#### VI. CONCLUSIONS

Flavor perception and stability are related to proteins, lipids, and carbohydrates as well as the conditions of storage. Although freezing offers an exceptional food safety and extended storage time, many reactions such as moisture migration, lipid oxidation, and protein denaturation promote off-flavor formation. Therefore stability can be achieved with a more integrated understanding of the flavor interactions within the complexity of foods.

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# 7

### **Food Sensory Attributes**

Patti C. Coggins

Mississippi State University, Mississippi State, Mississippi, U.S.A.

Roberto S. Chamul

California State University, Los Angeles, Los Angeles, California, U.S.A.

#### I. INTRODUCTION TO SENSORY EVALUATION

To measure sensory properties and thus determine if the overall objective was achieved is and will continue to be a major accomplishment for sensory evaluation. The development of knowledge and the achievements made possible through this gain regarding the attributes of a product or other pertinent information is another great source layered upon current sensory research and its general knowledge base. The capability of researchers and industry to perform sound sensory science techniques is at an all-time high. Growth in the area of sensory science has been exceptional during the last decade. Newly applied techniques and the development of new modes of observation and data retrieval continue to be implemented and are thrusting sensory science continuously further.

The measurement of human behavior is now directed at a more systematic and professional approach to the testing process. Much of the prior accomplishments were achieved from an indirect method either through marketing services, through relations to new product development, or through the dedication of a few select individuals devoted to the sensory profession. Advances in this field were initially slow due to the lack of interest and dedication in sensory science. Today, sensory science is a leading research tool applied to many processes and products.

The importance of the relationship between reliability and validity of results and the credibility of proposed recommendations is often a challenging process in sensory evaluation. Often, evaluations are performed, data is obtained, and results generated, which in turn are shuffled into the "meaningless" grouping due to the misunderstandings of and between the separate working group sectors within an organization. Tactics such as these decrease the viability of the sensory group performing the procedures because the achieved information was not properly reported. The success of sensory evaluation, in particular its credibility, begins with an organized effort, and the assurance that the testing process followed accepted procedures and practices in all respected areas of work.

The field of sensory evaluation, being a relatively new discipline, has provoked many divergent opinions and philosophies from both within and outside its parameters. It would be difficult to state the one or two progressive developments that were initially responsible for the emergence of sensory evaluation as a discipline and the limited base of acceptance

that ultimately defined the discipline. Agriculture and the international branching of it that began in the 1960s has been seen as one of the major pivotal areas providing growth and interest in sensory evaluation as a necessary function. Also, internationalization of the marketplace as a whole has greatly influenced this growth and interest.

The formal definition of sensory evaluation can be expressed in several different ways. However, the Sensory Evaluation Division of the Institute of Food Technologists (1,2) has expressed a definition that provides meaningful insight into the subject:

Sensory evaluation is a scientific discipline used to evoke, measure, analyze and interpret reactions to those characteristics of foods and materials as they are perceived by the senses of sight, smell, taste, touch and hearing.

This definition makes it clear that sensory evaluation utilizes all senses in determinations about products. Often, only taste is of consideration, and the industry tends to overlook that sensory evaluation is not just "taste testing." Also, this definition emphasizes the many disciplines that are involved with the complete and correct workings of sensory evaluation. Sensory evaluation is a quantitative science in which numerical data are collected to establish specific relationships between product characteristics and human perceptions (3). Sensory evaluation is a science of measurement. Like other analytical test procedures, sensory evaluation is concerned with precision, accuracy, sensitivity, and avoiding false positive results (4). Analysis of data is a critical part of sensory evaluation. Data generated from subjects are often highly variable. There are many sources of variation in individual responses that are extremely difficult to control in sensory testing. Examples include the participants' individuality in personality traits, habits, attitude, acuity of perception, product familiarity, and frequency of consumption.

The interpretation of results is one of the most difficult parts of the formal sensory evaluation process. Once the data are obtained and results generated, it is the tedious job of the sensory analyst to write the report carefully and supply the information to the necessary party of interest. Many persons housed in separate departments within a business or research facility take this information and focus on it in an incorrect manner owing to the outcome oftentimes not being as they would have liked.

#### II. PRINCIPLES OF SENSORY EVALUATION

The sensory technique chosen for a particular test is determined by the objective of the study. When testing for acceptability, trained panels should not be used; consumer groups should also not be used to generate descriptive data. Product samples should be labeled with three-digit random codes to alleviate bias. The sample order should be randomized to assist in reducing biases. Panelist influences should be kept to a minimum during evaluation.

#### A. Sensory Perception

Sensory adaptation, due to continuous exposure of a particular stimulus, will lead to a significant decrease in responsiveness. Foods can be complex mixtures encompassing all attributes. Due to the complexity of a mixture, suppression within modalities will occur. Thus the perceived intensity will be affected and could possibly be recorded as a lower value than if the intensity were to be recorded as a single attribute, which would represent a truer value describing intensity.

Taste and smell can be confusing. Many people cannot distinguish between the two owing to olfactory sensations arising from volatiles of the food matrix. If subjects are untrained, these sensations can be recorded incorrectly as either representing taste or smell inappropriately.

#### **B.** Sensory Testing

Sensory testing was initially developed from an industry standpoint to perform an economic function. It can determine the worth or acceptability of a product as its final result. The principal uses of sensory evaluation are in research, product development and maintenance, quality assurance, and as a marketing tool. Sensory testing encompasses many fields other than food. Its primary function is to conduct valid and reliable tests that will in turn provide necessary data upon which sound business decisions can be made.

If sensory analysis is to be dependable, the skill of the sensory analyst in optimizing the proper techniques in sensory testing is of optimum importance. There are several general steps that should be followed in sensory testing. The first step is to define the problem, followed by method choice, decision on panel type, experimental design, data analysis, and report generation. The method selection can be a decisive decision. The three categories of sensory techniques are discrimination procedures, descriptive analysis, and affective testing. The discrimination procedures are designed for difference detection; descriptive analysis is focused on sensory changes; affective testing gathers consumer, preference, or hedonic data.

The selection of the subjects who will be the measuring instruments of the designated study is a critical step in sensory testing. Humans are excellent relative measuring instruments but poor absolute measuring instruments. The basis of qualification of human subjects can be one of the most time consuming sections of the sensory project. Subject or panel maintenance, performance measurement, participation monitoring, and other concerns are necessary duties that must be continually kept in the forefront of the sensory study.

#### III. SENSORY EVALUATION OF FROZEN FOODS

Before frozen food can be evaluated properly, quality measurements must be determined and properly defined. The definition of the sensory quality of a food or food product is "the acceptance of the sensory characteristics of a product by consumers who are regular users of the product category, or who comprise the target market for the product" (5).

The effects of prolonged storage of foods in the freezing range are varied. In some instances, such as butter or fatty foods, the odor may become unpleasant by a process such as rancidity. In other foods, such as peaches, the process of oxidation may alter the appearance or color. In some products, such as fish, off-flavors may develop by both physical and chemical processes. In other cases, such as asparagus, the texture may become undesirable. And in many cases, the nutritive value of the fruit or vegetable may be affected by processes of hydrolysis and oxidation. All these criteria that go into food quality—odor, appearance, flavor, texture, and nutritive value—are important in the defining of "satisfactory" food storage.

For example, frozen peas will lose their desirable bright green color and acquire a yellow to brown tinge at temperatures above 15°F (6), owing to a slow destruction of

chlorophyll, the characteristic pigment of green vegetables and other plants. This change is not evident when the storage temperature is maintained at 0°F or less.

Yet tasting panel tests on frozen and stored halibut (7) and salmon (8) have clearly demonstrated that the temperature of storage is more important in maintaining quality than is the freezing rate. Even the texture of twice-frozen foods cannot be the equal of fresh-frozen quality produce. The following sections are brief overviews of selected foods and food groups. The authors selected the most predominant sensory aspects as the primary focus of discussion and review.

#### A. Water

The Safe Drinking Water Act (SDWA) establishes regulations for maximum contaminants (primary and secondary). It is not the aim of this section to review primary contaminants that may affect the health of consumers, but to review the secondary contaminants that can cause flavor problems in water itself and/or in products in which water with such defects are used. The International Association of Water Quality (IAWQ) has developed a Drinking Water Flavor Wheel that includes the four basic tastes recognizable by the tongue (sour, sweet, salty, bitter), and eight odor descriptors (chemical, medicinal, fishy, fragrant, swampy, grassy, woody, chlorinous). Mouth feel is a descriptor considered as part of the flavor component (9).

Table 1 shows the secondary contaminants in water according to the Environmental Protection Agency (EPA). Major problems encountered are the metallic taste caused by copper, iron, manganese, and zinc. A salty taste is caused by chloride, sulfate, and total solids dissolved. A bitter taste is produced by foaming agents, manganese, and low pH (10). Iodinated trihalomethanes, which are disinfection by-products, are considered to

**Table 1** USEPA<sup>a</sup> National Secondary Drinking Water Contaminant Standards

Contaminant	Effects	$SMCL^b mg/L$
Aluminum	Colored water	0.05-0.2
Chloride	Salty taste	250
Color	Visible tint	15 color units
Copper	Metallic taste, blue-green stain	1.0
Corrosivity	Metallic taste	Noncorrosive
Foaming agents	Bitter taste, odor	0.5
Iron	Metallic taste, orange staining	0.3
Manganese	Bitter metallic taste	0.05
Odors	Musty or chemical smell	3 TON <sup>c</sup>
рН	Low pH—bitter metallic taste, high pH—soda taste	6.5–8.5
Sulfate	Salty taste	250
Total dissolved solids	Salty taste	500
Zinc	Metallic taste	5

<sup>&</sup>lt;sup>a</sup> USEPA—U.S. Environmental Protection Agency.

Source: Ref. 10.

<sup>&</sup>lt;sup>b</sup> SMCL—secondary maximum contaminant level.

<sup>&</sup>lt;sup>c</sup> TON—threshold odor number.

cause medicinal odor with odor threshold concentrations of 0.1 to 8.9 g/L. Using flavor profile analysis (FPA), these odorants are described as sweet, solvent, and medicinal (11).

To control taste in water, it is necessary to control inorganic ions. However, malodorous organic compounds such as 2-alkyl-5,5-dimethyl-1,3-dioxanes and 2-alkyl-4methyl-1,3-dioxolanes have been identified in groundwater as well as in river water and tap water (12). The use of threshold odor number (TON) for odor detection in water seems not to be the best method, so the use of flavor profile analysis (FPA) has been suggested (13, 14). Some of the most common odor reference standards used in raw and finished drinking water for FPA are geosmin (earthy), 2-methylisoborneol (earthy), trans-2-cis-6-nonadienal (cucumber, green vegetation), *m*-xylene (sweet solventry), benzofuran (mothballs), cis-3-hexe-1-ol (fresh grass, green apple), and dimethyl sulfide (septic, musty) (15).

However, especially for drinking water samples from river sources, there may be 200 or 300 volatile organic compounds present at concentrations greater than 1 ng/L as measured by capillary gas chromatography (GC). Any of these chemicals alone or in combination can cause water sample odor. The odor-causing compounds are often unknown. The concentrations of individual compounds are usually below their odor threshold concentrations (OTCs). Since the identification of all the GC peaks of a typical drinking water sample by GC/MS is usually not possible, and the consideration of all compounds is desirable, statistical correlation methods can be used to describe mathematically the relationships between volatile chemicals and tastes and odors (16).

In the food industry, secondary contaminants have a zero tolerance for turbidity, color, odor, and taste. To achieve these criteria several water treatments are available. The traditional water treatment is coagulation/flocculation. An alternative is the multiple barrier treatment that includes the use of ultrafiltration/reverse osmosis.

#### **B.** Bakery Products

Baked goods are highly perishable, and their attractiveness declines rapidly within a few hours of being taken from the cooking mechanism. Freezing is the most used preservation method that significantly retards quality changes. This is the main reason for the huge market for frozen bakery goods. The history of the frozen food market began with frozen pizzas and doughnuts. The market has expanded tremendously in the last decade. Table 2 shows the most common sensory attributes of bakery products.

Like all foods with a significant level of water activity ( $a_{\rm w} > 0.9$ ), baked foods are subject to microbial contamination and growth during storage. Freshly baked bread is sterile; contamination with mold spores is deposited on the bread surface during cooling and/or packaging. Staling is another limiting factor in the shelf life of baked goods. During baking, the starch granules present in the flour gelatinize. Upon cooling and storage at ambient temperatures, this gelatinized starch slowly recrystallizes. This retrogradation contributes to an increase in crumb firmness, and leads to a harsh, dry mouth feel when the product is eaten (17).

Freshly baked product that has been properly formulated, frozen, stored, and refreshed (regardless of the time it has been in the freezer) shows sensory properties comparable with the same unfrozen product less than 1 day after baking (17). Quality defects observed in frozen, thawed, fully baked products can be traced, for the most part, to migration of water during freezing, storage, and thawing. Defects of migrational sorts are slimy icing, disappearance of icing, wet surface area. Staling is accelerated at low temperatures, which can often lead to toughness of product. This toughness is mostly due to denatured protein. A high-gluten flour gives a tougher product than a low-gluten flour.

**Table 2** Sensory Attributes in Frozen Bakery Products

Product	Attribute	
Bread	Crumb firmness	
	Harshness	
	Dry mouth feel	
Icing, glazes	Wetness	
	Leathery	
	Unappetizing	
Croissants	Flakiness	
	Chewiness	
	Wetness	
Pizza	Sogginess	
	Wetness	
Cakes	Crumbliness	

Source: Ref. 17.

The icings, glazes, and fillings of baked goods depend on a fine balance between water, sugars, and hydrocolloids to achieve the desired product characteristics (17). These properties include a dry appearance coupled with flexibility and a short body, no free water, and good suspension of solid fruit pieces for fillings. Freezing can alter the interaction between water and hydrocolloids so that on thawing, free water is present; this results in melting or disappearance of icings and glazes and leads to wet, leathery, unappetizing fillings. Ice crystals formed from free water are prone to sublime during frozen storage, and a rim of ice on the inner surface of the packaging indicates that the water has not been adequately bound and stabilized in the icing or filling (17).

The sensory properties of bread and rolls can be greatly altered by the handling of the products. The importance of complete cooling is utmost and greatly affects the quality. Bagels have a chewy texture, and improper freezing techniques can lead to a leatherlike texture. Also, soggy dough is a problem with bagels. Croissants are made with layered dough that is rolled into a thin sheet that is spread with shortening or butter. The detrimental freezing attributes here are decreased flakiness, chewiness, and often wetness. Pizza dough is ideally suited for frozen storage. Having a high surface-to-mass ratio, the dough freezes quickly and efficiently in a freeze tunnel. Waffles and pancakes are much like bread and rolls regarding proper handling techniques before freezing. Sogginess and wetness is often a problem with waffles and pancakes.

Cakes and other such sweet products have been successfully frozen without loss of sensory attributes. However, it has been noted that loss of volume by as much as 20% during frozen storage has occurred with certain formulations (17). Moister cakes showed better volume retention, whereas dry crumb cakes such as chocolate tend to develop a crumblier texture after being frozen. Pastries that are frozen develop a dryness and hardness. If they are filled with filling, the filling will often be gluey or rubbery.

Biscuits, due to the low moisture content, are successfully frozen for lengthy periods of time. Freezing of biscuits imparts flavor retention and is an excellent method for preserving baked goods of this type. Cookies or cookie dough have properties similar to those of biscuits when frozen. Products of this type have very good retention of sensory attributes when thawed.

Pies are handled very similarly to cakes, but they suffer a greater number of detrimental affects when thawed. Syneresis can be a major problem with pie that has meringue, or certain fillings. The fillings of crème pies can develop a rubbery or gluey texture after extended frozen storage and improper handling techniques. The filling must be formulated to withstand freeze—thaw cycles and still retain stability. Custard-type pies freeze well and typically thaw well with minimal loss of sensory attributes.

#### C. Dairy Products

The changes that occur during freezing vary among dairy products from minimal to extremely severe. The lower the temperature at which the product is stored, the slower is the deterioration rate of the product and the better the product quality after defrosting. Since the storage temperatures depend on the type of product (frozen or nonfrozen), there is a different relationship between the product in question, the storage temperature, and the product quality. Freezing points of dairy products should not be used as the temperatures at which to freeze the products. This is because storing products at a temperature above which ice is formed may successfully attain preservation in some cases. As a result, one needs to understand the impact frozen storage has on product quality before choosing freezing as a preservation method. For a number of dairy products the changes during freezing are minimal, while for others the changes are very serious. In liquid dairy products, freezing causes destabilization of proteins and fat. The destabilization (dehydration) of the protein is accompanied by an increased concentration of mineral constituents and lactose in the unfrozen part. Ice crystals and fat crystals contribute to the destruction of the fat globule membrane leading to the formation of free fat. The lower the temperature, the slower the speed of the deteriorative reactions and the better the quality after defrosting. For every dairy product there is a different relationship between the storage temperature and the time it takes to undergo a certain amount of quality change (18, 19). Table 3 provides a listing of common sensory attributes of frozen dairy products.

 Table 3
 Sensory Attributes of Frozen Dairy Products

Product	Attribute	
Fresh milk	Sweetness	
	Off-colors	
	Oxidized flavors	
	Lumping	
	Gelation	
Cheese	Color intensity	
	Odor intensity	
	Flavor overall, oxidation	
	Off-flavor	
	Texture—structure, crumbly	
	mealy, grainy, hardness	
Ice Cream	Texture—coarse, greasy	
	sandy, gritty	

#### 1. Milk

Fresh milk may be frozen if storage is required for consumption at a later date or if shipment of the product is required. When thawed the product should still taste relatively fresh. The flavor of unfrozen whole milk is pleasantly sweet, possessing neither a foretaste nor an aftertaste other than that imparted by the natural richness (20). In addition, it should have no off-colors or lumps visible to the eye. These attributes are often retained better through freezing than through any other method. However, if raw or pasteurized milk is improperly frozen, flavor deterioration may occur. Oxidized flavors may appear as a result of the destruction of the milk-fat globule membrane (21). These flavors may increase in intensity the longer the product is frozen. Tressler et al. (22) stated that the occurrence of the traditional oxidized flavor defect can be reduced by adding small amounts of ascorbic acid, pasteurizing at relatively high temperatures, homogenizing, and controlling the daily ration of the dairy cow.

Preservation of milk by deep-freezing also causes changes in the physicochemical balance, and separation of milk solids occurs upon thawing. Freezing milk at  $-10^{\circ}$ C results in the destabilization of proteins and fat, whereas storage of milk at -30 to  $-40^{\circ}$ C aids in retaining milk's stability longer (23). This destabilization, in turn, increases the mineral constituents and lactose present. By increasing the concentration of these components, tricalcium phosphate is precipitated and the pH value of the milk is reduced. Fat globule aggregation occurs, and milky layers and butterfat lumps are noticed (23). Milk fat and total solids are lower in frozen milk samples. Therefore milk should be stored at lower storage temperatures to minimize the separation of the milk components and increase the quality of the product. Milk fat and total solids have been shown to be lower for frozen samples than for those analyzed fresh. The Babcock fat test was also affected by the rate of freezing and possibly by an interaction between freezing and thawing rates. Milk fat, protein, and total solids in fresh milk can be accurately predicted from determinations on milk that had been frozen and thawed (24).

Thawing of the frozen milk is also critical in minimizing sensory defects. Thawing the container of frozen milk in warm water or in a refrigerator overnight are considered satisfactory methods with minimal adverse defects (22). However, if the product is thawed improperly and lumps or gels are noticed, warming and stirring the milk may aid in redispersing the protein.

#### 2. Cheese

Quality and acceptability defects from freezing are probably most noticed in cheese. Cheeses vary based on the type of milk they are made from, the method of milk coagulation used, the amount of whey retained in the curd, whether the curd is ripened or unripened, the method of ripening, and the type of milk used to make the cheese (goat, cow, sheep). Structure and quality of the cheese are greatly affected by the fat content. The variances among cheese types affect the overall color, texture, odor, and flavor of the product. Freezing is not usually recommended with ripened cheese as a preservation method owing to the physical breakdown in the body and structure of the product. However, it is useful in preserving unripened cheese like cottage curd. Previous studies have indicated no differences in the variables between the control and the frozen cheeses. The increase in pH at the end of ripening was less pronounced in the frozen cheese but not significant.

Processed cheese does not freeze well even with the incorporated emulsifiers typically found in these types of cheese foods. Gordon et al. (25) stated that on no account is

processed cheese to be stored below freezing point since freezing has a deleterious effect on the cheese structures as in normal cheese. The appearance of structural defects in processed cheese depends on the temperature, the concentration of water-soluble substances in the cheese, and its fat content. The physical change of the structure of frozen cheeses does not affect the subsequent quality of the processed cheese manufactured from them. In a time of surplus, Gouda and Cheddar cheese can be stored for 1 year (or even longer) at  $-10^{\circ}$  to  $-20^{\circ}$ C and can then be used for the manufacture of a good quality processed cheese. This method of preservation prevents the development of off-flavors and unpleasant sharp flavors found in cheese stored for a long time at normal ripening temperatures. According to Burkhalter (26), Emmental and Gruyère cheese used for making processed cheese may be stored frozen at  $-20^{\circ}$ C, but otherwise untreated, for approximately 18 months, provided that the outer zone (rind) with its tallowy, soapy flavor is removed. Storage at  $-10^{\circ}$ C for 5 months had no significant effect on the structure and quality of processed cheese, while the same type of cheese stored at  $-30^{\circ}$ C became mealy. A cheese spread was even stable at  $-30^{\circ}$ C.

Storage experiments carried out on skim milk cheese and cream cheese at  $-10^{\circ}$ C and  $-25^{\circ}$ C showed that these cheeses could be stored for up to 6 months. Apart from a slight whey separation after thawing, the texture remained smooth and the flavor was good. American cottage became somewhat softer and had a rancid and sharp flavor. The off-flavor was probably due to the poor quality of the cream used for dressing. The results indicated that cream cheese can successfully be stored at  $-20^{\circ}$  to  $-30^{\circ}$ C for 4 to 12 months depending on the initial quality of the cheese (23).

External characteristics of cheese are the first items noticed by a consumer and qualified judge. The color is less intense in frozen cheese. Luck (23) stated this might be due to the microstructural changes of the paste, which occur due to the formation of ice crystals resulting from freezing the product. The structural changes give rise to a more open structure increasing the refraction and clarity of the cheese. In addition, the number, size, and distribution of eyes formed in Swiss cheese are reduced and irregularly distributed.

Consumers observe the internal body and structural characteristics next, which include the texture and flavor of the product. This is where the major defects occur in cheese that is frozen. The physical structure of the cheese is broken down during the freezing and thawing stages. The extent to which the structure and body are affected depends on the degree of ripening, the amount of salt used in the formulation, and the moisture content in the cheese variety. This in turn affects the degree to which ice crystals are formed when the cheese is stored between  $-1^{\circ}$  to  $-16^{\circ}$ C and the degree to which the defects are noticed. Hydrogen bonds in the polypeptide chains break down, causing water to separate from the protein. A crumbly and mealy body is produced. The grainy texture of the body of cheese is intensified by slower freezing speeds (27). The main defect of cheese made from frozen curd is in body and texture. The external appearance of cheese pieces is usually normal, whereas internally they were made up of layers, similar to those observed by Schulz in soft cheeses. This layered structure may be from ice crystal formation in the serum phase between the casein micelles, although the deep-freezing was rapid (30 minutes at  $-40^{\circ}$ C). The method used in France for deep-freezing curd is only for hard cheese. In this case mechanical stirring of curd or its mixing with freshly made curd before further cheese making, can be used to reduce this texture defect. The cheese produced is not inferior to the cheese made under normal conditions in constituents or sensory qualities. Deep-freezing of curd is applicable for making Teleme from ewe's milk (28).

The odor intensity and overall taste and acidity are also modified by freezing and storage. An increase of hardness and decrease in creaminess in frozen cheeses after 3 months of storage has been reported (29). However, the acidity, odor, flavor, and intensity decrease during storage (29), producing a very mild cheese. Frozen storage did not result in significant alterations in overall compositional, rheological, and sensory properties or the level of lipolysis. The extent of proteolysis was slightly lower in the cheeses frozen prior to ripening, although, some satisfactory results have been achieved when freezing soft cheeses (30).

The grainy aspect of cheese can be significantly affected by storage time. Quality parameters remained relatively constant up to 180 days storage, typically followed by sharp increase thereafter. This increase is a clear sign that the texture of the cheese is considered altered and granulous properties therefore determined. The graininess of cheeses frozen at slow speeds and for 9 months have been shown to be significantly different from that of the other storage methods for cheeses. Cheese graininess is therefore more intensely affected at slower freezing speeds and when the frozen storage period exceeds 6 months. In this respect, Luck (23) reported that freezing caused a breakdown in texture, prompted by the formation of ice crystals and the subsequent alteration of proteins.

Although some authors have described changes in the texture of frozen cheeses, it is recognized that the intensity is greater with slower freezing processes (27). Neither the storage time nor the freezing process significantly modified the hardness and creaminess of the cheeses during storage. Slower freezing processes should be employed to minimize the flavor, odor, aroma, and textural defects when processing cheese. The changes of the texture due to freezing can be prevented by storing the cheese at temperatures just above its freezing point  $(-2^{\circ} \text{ to } -5^{\circ}\text{C})$ . This is a successful method of preservation. Camembert cheeses frozen immediately after making were acceptable after defrosting under normal conditions for up to 3 weeks. Not only does a slower freezing process minimize quality defects, it is more economical from the industry's viewpoint. When the freezing process is employed in this manner it is a practical and economical method in regulating the quality of cheese products, especially that of ewe cheese (29). Consequently, once the cheeses have been frozen, these attributes would not be modified by excessively long storage times. In this respect, Diefes et al. (31) concluded that freezing led to local protein dehydration, owing to the destruction of the protein matrix, giving rise to greater hardness of cheese. Differences are typically not seen during more prolonged periods of frozen storage in terms of the attributes characteristic of the appearance of the paste, color, and eyes, owing to the specific freezing procedure used. Odor, flavor intensity, acidity, and grainy appearance change as a consequence of frozen storage. With up to 6 months storage, practically no differences can be detected with respect to the nonfrozen cheeses; however, after 9 months storage, the acidity, odor intensity, and flavor decreased, and the grainy body increased substantially. Saltiness was not affected by freezing nor by frozen storage. The speed of the freezing process only affects the graininess of the cheese, which is slightly greater in slowly frozen cheeses. From the sensorial standpoint, freezing is therefore a suitable method for regulating the cheese market.

Cheeses could be stored at  $-20^{\circ}$ C for at least 3–6 months, ensuring their availability for sale throughout the year. Slow freezing speeds are recommended, since the small deficiencies in quality detected in cheese frozen at slow speeds are recovered from an economic standpoint, since the faster process is much more costly. Alterations in texture (28, 27) and the presence of oxidized flavors were the main problems detected in cheeses after thawing. However, some of these studies have not reported any deficiencies in frozen

cheeses (32); occasionally a higher sensorial quality is obtained with respect to nonfrozen cheeses (33). Odor intensity, intensity taste, and acid taste were modified by freezing and storage. Significant differences have not been detected between salty tastes of nonfrozen cheeses and frozen cheeses and those stored in freezing.

Texture is substantially altered by freezing and frozen storage, with noticeable increases in the grainy body at 9 months of frozen storage. In the same way, an increase of hardness had been detected and a decrease in creaminess in frozen cheeses from 3 months of frozen storage. The external characteristics of the cheeses also demonstrate important changes after freezing. Color is less intense in frozen cheese. This could be due to microstructural changes in the paste, produced by the formation of ice crystals, giving rise to a more open structure, increasing refraction and making the cheese seem clearer. The number, size and distribution of the eyes are reduced after 90 days of storage in freezing, probably owing to the destruction of the structure or wall forming the eyes. These usually decrease in size and in some cases disappear, giving rise to a more irregular distribution. Even though salty flavor is not modified by either storage time or the freezing process used, the intensity of flavor decreases significantly with longer frozen-storage times. Flavor intensity remains practically stable over up to 6 months frozen storage, decreasing rapidly thereafter, and regardless of the freezing process used. Some authors have reported no differences in terms of flavor intensity between cheeses prepared from frozen curds and unfrozen cheeses (33), although others have also described very low scores in slowly frozen cheeses when thawed immediately (34). Similarly, other authors have reported no modification in the flavor of Cheddar cheese undergoing frozen storage (35).

As with the intensity of acidity, frozen storage time has a significant influence on the acidity of the cheese. Texture alteration is one of the adverse effects of freezing in cheese. This quality defect has customarily been attributed to the formation of ice crystals and the resulting mechanical effects. However, other findings suggest that alterations in cheese texture are a consequence of changes in protein structure, such as breaking of hydrogen bonds in the polypeptide chains and changes in water binding capacity. Such changes lead to more active proteolysis in cheeses that have previously been frozen. In prefrozen cheeses, unordered structures increase immediately after thawing. The proportion of unordered and turn structures is apparently higher in the cheeses frozen more slowly in a plate freezer than in the cheeses frozen more quickly in liquid nitrogen vapor (27).

Slower freezing processes should be used to minimize the flavor, odor, aroma, and textural defects when processing cheese. The changes of the texture due to freezing can be prevented by storing the cheese at temperatures just above the freezing point  $(-2^{\circ}$  to  $-5^{\circ}$ C). This is a successful method of preservation. Camembert cheeses frozen immediately after making were acceptable after defrosting under normal conditions for up to 3 weeks. Not only does a slower freezing process minimize quality defects, it is more economical from the industry's viewpoint. When the freezing process is done in this manner, it is a practical and economical method for regulating the quality of cheese products, especially that of ewe cheese (29).

#### 3. Ice Cream

Freezing is the standard method for processing and preserving ice cream. The quality, palatability, and yield of the finished product are all dependent upon freezing of the ice cream mix. Since no two individuals will execute the details of the freezing operation in exactly the same way each time, programmed freezers are becoming widely used in the industry to control the quality of the product. An ideal ice cream possesses a fresh, clean,

pleasant, and delicate flavor with a smooth texture. It has a natural color; melts slowly into a liquid state, and any particulates present are evenly distributed (36). Regardless, however, of freezer automation, defects can still occur in the flavor, body, and texture of the product.

Texture defects are the most common result of improper freezing of ice cream. The most frequently noticed texture defect is referred to as coarse. It is observed by a grainy or icy feeling and accompanied by unusual coldness. Numerous factors contribute to this widespread defect. Higher fat mixes decrease the occurrence of this defect in the product because fat globules obstruct the growth of ice crystals. Dipping cabinets are usually kept around  $-15^{\circ}$ C. At this temperature it is reported that ice crystals can double in size, increasing the coarseness of the ice cream (36). Therefore temperature control in dipping cabinets should be more closely regulated to minimize this particular texture defect. A greasy mouth feel is often associated with the buttery defect. The cause is over agitation of the ice cream mix in the freezer and can be reduced through automated freezing operations. One of the most objectionable defects in ice cream is known as a sandy texture. A gritty mouth feel results from crystallization of lactose when temperature fluctuations and extended storage times occur. Again, temperature control is critical in controlling texture defects in ice cream.

Flavor and body defects usually result from the addition or absence of ingredients. However, one body defect resulting from temperature fluctuations during storage is that known as shrunken. The ice cream product does not contact fully the sides of the container according to Marshall and Arbuckle (36). When products are frozen to unusually low temperatures, or if the product is allowed to warm and then is refrozen, the pressure on air cells changes and results in a shrunken appearance not desired by consumers.

# 4. Yogurt, Cultured Milk, and Buttermilk

Normal yogurt, cultured milk, and buttermilk are not successfully frozen to prolong their storage life. After a storage period of 3 months at  $-10^{\circ}$ C the flavor is satisfactory but the texture is completely broken. There is an intense whey separation and the products give a mealy sensation on the tongue. Stirred yogurt may be successfully frozen ( $-26^{\circ}$ C) provided that the total solids content of the yogurt is sufficiently high as in the case of plain yogurt. Storage experiments carried out at  $-25^{\circ}$ C on yogurt of higher solids content did not confirm these results. The texture of the defrosted products was only slightly improved. The quality of the yogurt was such that it would hardly have a chance on the market. Because it is possible to make frozen yogurt dessert, it will probably be possible in the future to preserve yogurt by freezing after adding a suitable stabilizer. At this stage fermented milks are not suitable for preservation by freezing.

### D. Fish and Seafood

Human perception of food is complex and may be influenced by the sensory characteristics of the food material but also by the personal experience of the observer, psychological state, ethnic origin, religious conviction (37). Analytical testing to determine differences or similarities in seafood, to identify quality attributes, or to estimate the relative intensity of sensory attributes is usually accomplished in a laboratory setting. Difference tests are designed to identify samples that differ in terms of sensory characteristics. Ranking tests (ordinal scaling) are used to determine the rank order of a set of samples for degree of a specified sensory attribute. Scalar testing methods are used to provide information about

quantitative relationships among product samples. Descriptive analysis is the most demanding of any sensory method. Panelists determine the qualitative characteristics of aroma, flavor, texture, aftertaste, etc., and they describe a set of descriptors. The traditional flavor profile method requires intensive training (38).

Changes in appearance and eating quality that occur during frozen storage of fish are of great commercial importance. Freeze burn appears on the surface of frozen fish due to loss of moisture, cooked muscle flavor may possess a characteristic "cold store" note, and toughness and dryness increase, rendering a less acceptable fish. Tasting and determination of sensory attributes still remains the method of choice to detect changes during frozen storage. Sensory methods used to assess frozen fish can be classified into three categories: (a) those in which tasters are asked to assess the fish on a scale of acceptability; (b) those in which certain diagnostic features are rated on intensity scales; and (c) those in which comparisons between two samples are made in order to distinguish the sample under test. Sensory factors that change during frozen storage include toughness, dryness, and flavor. Scoring systems based on odor, texture, and flavor have been developed for fish inspectors. Freezing and frozen storage affect the appearance of fish on thawing (39).

The first quality judgment made by a consumer about a fish product at the point of sale is based on its color/discoloration. The next judgment is about the product's texture: any gaping or ragged fillets. This is followed by odor judgment. A standard sensory evaluation for fish should include color of the flesh, odor and visual texture, absence/presence of blood clots and of bruising and discoloration, absence/presence of physiological abnormalities, and workmanship (40). Aroma is one of the most important freshness determinants of fresh fish and fishery products. Sensory assessments are desirable because they provide immediate quality information. Initial changes in the aroma of freshly caught fish involve the shift from the fresh, planty aromas to a neutral, flat-sweet odor. This characteristic sweet aroma appears to be contributed by alcohols and 3,6-nonadien-1-ol, which has a sweet melonlike aroma (41).

Frozen storage is an important preservation method for seafood. Freezing and frozen storage contribute to changes in texture as a result of protein denaturation. Also, the thawing method is important, since it is recognized that this can affect the sensory attributes of seafood. Myofibrillar proteins undergo denaturation and aggregation during frozen storage. Different species of fish have different degrees of susceptibility. Ingredients such as triphosphate, lecithin, and sucrose ester have been used in order to prevent those changes that will affect the textural properties of muscle (42).

Considering all foods under the category of seafood, there is a large range in texture. As long as texture falls within the normal range, it does not influence preference as do flavor and appearance. Texture is a critical factor for shellfish, surimi-based products, and squid (43). The texture of fish muscle changes from soft, moist, and succulent to unacceptably firm, hard, fibrous, and dry. The unacceptability of fish flesh/muscle as food is caused by its increased tenderness due to myofibrillar fragmentation. At -8 and  $-20^{\circ}$ C, carp shows degradation in its myofribrillar fraction after six months (44). Freeze—thaw cycles affect the loss of protein solubility and the physicochemical and enzymatic properties of cod muscle proteins (45).

Sensory analysis is used to assess the quality of fish products. However, it is considered subjective and prone to error. During frozen storage there is an alteration of functional properties of muscle proteins, loss of water-holding capacity, juiciness, and changes in texture. Mechanical properties can be measured. Shear resistance can be measured using the Kramer shear-compression cell. Correlation between sensory analysis and shear resistance measured by Kramer has been reported in fish hardened as a result of

time in storage and also in fish stored at different temperatures. Shear resistance can also be measured using the Warner-Bratzler shear cell. Shear resistance as analyzed by the W-B cell has been found to increase significantly with time in frozen storage under different forms of preprocessing. The puncture test consists of measuring the force required to push a plunger into a food sample. This method assesses changes in texture during storage in ice as associated with the onset of rigor mortis. Tension analysis measures the force required to break a sample when is held by two parallel clamps. This test is sensitive enough to measure the viscoelastic properties of fish muscles and has been used to quantify textural changes in the mantle of squid. In compression analysis, the deformation is measured upon application of compression force. This analysis can be performed in one or two successive compression-relaxation cycles. In minced or cooked fish products, a deformation of 50% is customary. However, for some mince products and fillets a deformation of 30 and/or 10% is used. Firmness generally tends to increase with time in frozen storage. Correlations between one-cycle compression measurements and sensory parameters such as elasticity in mackerel (Scomber scombrus) and hake (Urophycis tenuis) have been reported. When the first compression-relaxation cycle is followed by a second compression cycle, the test is known as texture profile analysis (TPA) (46).

Common descriptor terms for cooked seafood include flavor intensity, fresh fish, gamey, old fish, sweet, briny, sour, seaweed, bitter, fish oil, buttery, nutty, musty, ammonia, metallic, shellfish, flakiness, firmness, moistness, chewiness, mouth-drying, fibrousness, oily mouth coating, cohesiveness, whiteness, and darkness (47). The Flavor Profile<sup>TM</sup> method is used to describe the aroma, flavor-by-mouth, and aftertaste attributes of cooked fish muscle. Intensity of aroma and flavor character notes are rated on the traditional 4-point profile scale: 0 = just recognizable or threshold; 1 = slight; 2 = slightmoderate; 3 = strong; a designation of 0.5 was used to show an intermediate level of intensity. To describe 17 species of North Atlantic fish the following aroma descriptors were used: briny, sweet, fresh fish, old fish, stale fish, sour, shellfish, gamey fish, fish oil, earthy, nutty-buttery, musty, and scorched. The flavor descriptors were: salty-briny, sour, sweet, fresh fish, old fish, stale fish, shellfish, gamey fish, fish oil, earthy, nutty-buttery, canned salmon, bitter, metallic, mouth-drying, and mouth filling (48). A new seafood nomenclature system, to be used on the sensory or "edibility" characteristics of fish, has been developed by the National Marine Fisheries Service (NMFS) of the U.S. Department of Commerce. The goal of this system is to enable consumers to make educated choices among novel species by providing the comparative sensory data necessary to select a desired flavor, texture, etc., of fish. Standardized methodology for evaluating the sensory properties of cooked fish has been developed (49).

# 1. Bluefish

Bluefish has a strong total aroma and flavor impact, with a highly aromatic distinctive gamey, fresh fish, sour, fish oil character. The amplitudes (overall impressions) are moderate, with character notes blending together to give a very full aroma and flavor. The aroma of bluefish has an early impact of a slight to moderate briny, moderate fresh and gamey fish notes, slight fish oil and sour notes, and a very slight sweet. The flavor has an early impact of slight sweet, moderate fresh fish, slight to moderate gamey fish and sour (sharp), very slight fish oil and salty-briny, and slightly mouth-drying. This fish has a distinctive sour (sharp) at a higher intensity than in most fish. In both aroma and flavor, the gamey and fish oil notes seem to be related. They are found at higher intensities in the dark flesh (especially near the skin) but are also present in the lighter flesh. The aftertaste is

strong and persistent. The reported Profile integrates proportionate light and dark flesh. The most common sample variation was in the intensity of flavor of the fish oil and sour notes, and sometimes the flesh and gamey notes (48, 49, 37).

#### 2. Blackback Flounder

Blackback flounder has a low total intensity aroma and flavor with a delicate, fresh fish character. The aroma amplitude is just below moderate and the flavor amplitude is moderate. The aroma is slight sweet and briny, slight to moderate fresh fish, and very slight sour. The flavor is slight sweet, slight to moderate fresh fish, below slight salt-briny, sour and mouth drying. The aftertaste is a low level fresh, mouth-drying and sweet (48, 49, 37).

### 3. Catfish

Frozen catfish (*Clarias gariepinus* Burchnell) fillets frozen and stored at  $-20^{\circ}$ C for 11 months using an oxygen-permeable packaging (OPP) material and a vacuum packaging (VP) showed no significant differences between any of the aroma, appearance, texture, or flavor attributes for the two packaging materials over time during frozen storage. Texture of the catfish fillets changed after 7 months, becoming more flaky with few ice crystals between the fiber, more so in the fillets packed in the OPP. No color changes were observed during the 11 months of storage. After this period of time the frozen catfish fillets were acceptable (50). In another study, channel catfish (*Ictalurus punctatus*) was evaluated during frozen storage, and it was concluded that regardless of size, channel catfish is acceptable after 12 months of frozen storage (51). On the other hand, channel catfish fillets treated with ascorbate and kept frozen at  $-6^{\circ}$ C for up to 6 months showed less rancid flavor (52).

#### 4. Cod

The flavor of cod (Gadus callarias L.) has been described as sour and slight cooked potato. Common flavor descriptors are buttery, green, biscuit-like, roasty, mushroom like, cabbage-like, fatty, and metallic. The malty flavor defect is caused by a strong increase of 3-methylbutanal (53). Cod (market) has a moderately low total intensity aroma and flavor, with a low sweet, fresh fish character. The aroma and flavor amplitudes are moderate, as the overall impression of cod is of a mild fish with an uncomplex aroma and flavor. The aroma is slight to moderate briny and flesh fish, and below slight sweet, sour and shellfish. The flavor is slight sweet and mouth-drying, slight to moderate fresh fish, very slight salty-briny and sour, and below slight shellfish. The aftertaste is fresh fish and sour. Occasionally, samples of cod have a stale fish character note. Cod (scrod) has a moderately low total intensity aroma and flavor, with a low sweet, fresh fish character. The flavor is below slight sweet, slight to moderate fresh fish, very slight salty-briny, sour, and earthy (slight cooked potato), and slight mouth-drying. The aftertaste is fresh fish, mouth-drying, and sour. Some cod (scrod) samples have produced a stale fish character note and/or a metallic note.

During processing and storage, fish quality may decline due to oxidation of unsaturated lipids causing off-flavors and odors. In lean fish, the production of formaldehyde (FA) is important along with protein denaturation and texture changes. Cod (lean fish) and haddock (non-FA-forming) show hydrolytic activity during frozen storage. Storage at  $-30^{\circ}$ C decreases hydrolytic activity (54). For cod, deterioration in

texture of the thawed product is a serious problem. The presence of formaldehyde due to enzymatic degradation of trimethylamide oxide (TMAO) into dimethylamine (DMA) and formaldehyde (FA) is an increasing factor of the denaturation of proteins. The quality index method (QIM) is normally used to evaluate fresh and frozen cod. This method is based on a selected number of independent parameters that describe the quality of thawed cod. The scores for all single parameters are added to give the total quality index. For fillets the parameters are texture, color, bloodstains, and gapping, which vary from 0 to 3, and odor and parasites, which vary from 0 to 2 (55).

Cod fillets are generally frozen after catch. It is recommended that fish filleted prior to *rigor mortis* must be frozen to diminish the risk of shortening and deforming. Cod fillets kept at frozen storage for 17 months and evaluated by a trained panel showed that fillets frozen before rigor keep fresher than those frozen after rigor after two months of frozen storage. The attribute "dry/succulent" is rated on a scale from 0 to 100. Cod receives a score between 60 and 70 after two months of frozen storage. After 12 months fillets are evaluated "drier" (score = 40). There is a direct relationship between frozen storage and the attribute succulent/dry. The same relationship has been reported for frozen storage and "tough/tender" (56).

North sea cod kept for 2, 5, 8, 14, and 17 days in ice, filleted and frozen in an air blast at  $-34^{\circ}$ C and stored at  $-14^{\circ}$ ,  $-22^{\circ}$ , and  $-29^{\circ}$ C showed a gradual development of unpleasant odor and flavor called "cold storage odor." At the same time the fish becomes firmer and drier until it becomes tough and dry. Freshness flavor is unaffected by the freezing and thawing treatment, while firmness and dryness increase significantly (57). During freezing at  $-20^{\circ}$ C, loss of tenderness and of water-holding capacity, and fish flavor changes, may occur. Prolonged storage of fish causes deterioration of texture as described in increased toughness, chewiness, rubberines, or stringiness. Toughness increases over time, whereas cohesiveness decreases during storage. Higher storage temperature results in faster rates of textural deterioration of cod muscle (58). Frozen cod fillets change texture over frozen storage (59) as a result of muscle protein denaturation causing toughness of the muscle. Rapidly frozen and slowly frozen cod show no more denaturation after one year of storage than newly frozen fillets (60). Sensory attributes of thawed and refrozen cod include color, flavor (strength, fresh fish, saltiness, sweetness, stale, rancid, bitterness, and other flavors), and texture (firmness, flakiness, resilience, juiciness, cohesiveness, fibrousness, roughness, and fiber size). Thawing/freezing cycles do not affect the structure of the muscle even after 9 months of storage. However, slow thawing followed by refreezing resulted in fish that was grayer in color and staler in flavor. Storage time affected the sensorial attributes (61).

Frozen cod can develop off-flavors during storage. The main component for this off-flavor is hept-cis-4-enal. Samples stored for 18 months at  $-15^{\circ}$ C had some of the following compounds: hept-trans-2-enal and hepta-trans-2,cis-dienal. These two compounds were examined and described as cold storage flavor (cardboard, musty). Threshold study showed that hept-cis-4-enal is detectable in very low concentrations (0.00004 ppm). Aldehydes present in cod samples originated from lipid oxidation and tend to increase with temperature of storage. The major aldehyde produced was hepta-trans-2,cis-dienal (62).

#### 5. Cusk

Cusk has a moderately low total intensity aroma and flavor, with a low sweet, fresh fish character. The aroma and flavor amplitudes are below moderate, as this fish lacks fullness and blending, especially in flavor. The aroma is below slight sweet, slight to moderate fresh

fish, slight briny and sour, and below slight shellfish. The flavor is below slight sweet and shellfish, slight to moderate fresh fish, slight salty-briny and mouth drying, and very slight sour. The flavor is flat, washes out quickly, and is affected by the chewy, slightly tough texture. The aftertaste is fresh fish, sour, and mouth-drying (48, 49, 37).

#### 6. Haddock

Haddock has a moderate total intensity aroma and a moderately low total intensity flavor, with a sweet, fresh fish, shellfish character. The aroma amplitude is just below moderate, and the flavor amplitude is moderate, as this fish has a fullness of flavor and an interesting shellfish characteristic. The aroma is slight to moderate briny, and fresh fish slight sweet, shellfish, and mouth-drying, fresh fish at slight to moderate, sour at below slight, and salty-briny at very slight. The sweet, fresh fish, and shellfish flavors seem to be related, and the sour taste is fleeting. Earthy (slight cooked potato) and metallic notes, particularly in the dark flesh portion, have been recorded. The aftertaste is fresh fish, sour, and shellfish. There was some variation within a session and between sessions in the haddock samples, particularly in aroma, and was evidence as stale fish character notes (48, 49, 37).

### 7. Hake

White hake has a moderately low total intensity aroma and flavor, with sour (dishrag) aroma, and an earthy note in flavor. The aroma and flavor amplitudes are low to moderate owing to the old fish, sour (dishrag), and earthy notes, which indicated a lack of blended characters. The aroma of white hake is a slight to moderate sour (dishrag) and fresh fish and below slight sweet and briny. Aroma notes mentioned by panelists are old fish, earthy (slight cooked potato), and shellfish (clam). The flavor is slight sweet, sour (sharp), and mouth-drying, slight to moderate fresh fish, below slight salty-briny and earthy (slight cooked potato). There was an unblended quality to the flavor, and when both earthy and old fish occurred, they seemed related. The aftertaste is fresh fish, mouth-drying, sweet, earthy, and sour. This fish often showed great variation in the fresh fish and old or stale fish characteristics.

Quality of hake species (Merluccius merluccius, M. hubbsi, and M. capensin) varies for many reasons ranging from inappropriate fishing and freezing methods to inefficient distribution. Textural (mechanical) analyses such as puncture test, Warner-Bratzler, and Kramer press have been used in order to account for the variance of frozen fillets. As a result of this, four clusters were identified, ranging from excellent quality (low texture, high viscosity) to very poor (high texture, low viscosity). The parameters required for this classification are viscosity, maximum force from Kramer, and maximum force and energy from the puncture test (46). Frozen Patagonian hake fillets (Merluccius hubbsi) evaluated by using the cook sensory assessment (CSA) shows that fillets remain acceptable for over 12 months both at -20 and  $-30^{\circ}$ C (63).

### 8. Halibut

Halibut has a moderate total intensity aroma and flavor, with a sweet, fresh fish character, and a very slight gamey fish flavor note. The aroma and flavor amplitudes are below moderate, as some samples lack a blended flavor. The aroma is slight briny, slight to moderate fresh fish, below slight sweet and sharp, sour (pungent). The flavor is very slight sweet, slight to moderate fresh fish, above slight sour, slight salty-briny and mouth-drying, below slight gamey and fish oil. The definite sour flavor note is higher than in most fish.

The aftertaste is predominantly sour and mouth-drying, with some fresh fish and another hard-to-describe note that seems to be a combination of fish oil and gamey (48, 49, 37).

### 9. Herring

Whole herring (Clupea harengus) wrapped in polyethylene film and block-frozen using plate freezers and frozen for 32 days at  $-24^{\circ}$ C were beheaded and salted. Ten trained panelists conducted a sensory analysis to evaluate taste using the following descriptors: ripened, raw, malty/creamy, stockfish, salty, sweet, spicy, and aftertaste. The attributes for texture were: softness, watery, and toughness. Spice-salted herring taste characteristics were typical with a dominant malty/creamy taste and a fairly low intensity of stockfish character. The thawed salted herring achieved higher values of the ripened taste during the first few weeks in comparison with the fresh salted herring. Also, the thawed spice salted herring appeared to become soft quickly during storage, indicating a faster rate of ripening. Hardness increased after salting but decreased steadily. Hardness of fresh spice-salted herring at 21 weeks was comparable with thawed herring kept for 16 weeks (64).

Herring is susceptible to lipid oxidation during processing and storage. Prefreezing storage in refrigerated seawater followed by minimal tissue disruption, removal of oxygen, fast freezing, and low freezer storage temperatures reduce rancidity and give the longest shelf life. Fillets of herring kept on ice prior to storage at  $-18^{\circ}$ C for up to 84 days show that samples, held for 6 days on ice, formed oxidation product at the highest rate during frozen storage. Ice storage had a greater impact than frozen storage (65).

## 10. Grouper

Grouper has a moderately low total intensity aroma and flavor, with a low level of aromatics. The aroma and flavor amplitudes lack full-bodied characteristics, have a low impact of blended character, and low intensity of fresh fish characteristics. The aroma of grouper has an early impact of slight sweet, and briny notes, and slight to moderate fresh fish, followed by a very slight sour (pungent), and a shellfish note. The flavor is very slight sweet, salty-briny, slight fresh fish, sour (sharp), and shellfish, and slight to moderate mouth-drying (the highest intensity for the fish tested). The aroma and flavor shellfish note has a clam character. There are confusing overtones to the sour and shellfish notes that are difficult to describe. The aftertaste is persistent and consists of a moderate level mouth-drying and sour, and a low level of fresh fish. The dry, tough texture is difficult to ignore. The samples seemed relatively consistent within a session, and the greatest variations between sessions seemed to be in the sour, shellfish notes, with confusing overtones (48, 49, 37).

### 11. Mackerel

Mackerel has a strong total aroma and flavor impact, with a highly aromatic distinctive gamey, fresh fish, fish oil character. The amplitudes are moderate, with character notes blending together to give a very full aroma and flavor. The aroma of Mackerel has an early impact of moderate gamey and fresh fish notes, a slight to moderate fish oil, and slight sweet, briny and sour notes. The flavor has an early impact of moderate gamey and fresh fish notes, a slight to moderate fish oil, slight sweet and sour (sharp), and a very slight salty-briny and mouth-drying. The mouth-drying characteristics seem largely overpowered by the oiliness. In both aroma and flavor, the gamey and fish oil notes seem to be related. They are found at higher intensities in the dark flesh, but are also present in the lighter

flesh. The reported profile integrates proportionate light and dark flesh. The gamey fish note of mackerel is heavy and sometimes associated with a burnt or scorched characteristic. Despite the intensity and heaviness of this fish characteristic, it is typical of fresh mackerel and not indicative of aged fish. The aftertaste is strong and persistent (48, 49, 37).

### 12. Monkfish

Monkfish has a moderately low total intensity aroma and flavor. Its distinguishing characteristic is a distinctive shellfish aromatic note similar to lobster and clam. The aroma and flavor amplitudes are moderate, as this fish is full, complex, and blended with interesting notes. The aroma is slight to moderate briny (fresh, seaweed), fresh fish and shellfish, and slight sweet and sour. The dark flesh and gelatinous areas near the skin have more intense briny-seaweed and shellfish-clam character than with flesh. The flavor is slight sweet and sour, slight to moderate shellfish and fresh fish, threshold salty-briny, and very slight mouth-drying. The shellfish note adds interest and complexity to the aroma and flavor. The sweet, shellfish, fresh fish and buttery notes seem closely related and give a full flavor. The aftertaste is sweet, fresh fish, and shellfish (48, 49, 37).

#### 13. Pollock

Pollock has a moderately low total intensity aroma and flavor with sweet, fresh fish, low shellfish character. The aroma and flavor amplitudes are moderate, as they are blended with some interest notes. The aroma is slight sweet, briny and sour (sharp), slight to moderate flesh fish, and below slight shellfish-clam. The flavor is slight sweet, sour (sharp) and mouth-drying, slight to moderate fresh fish, below slight shellfish-clam, and very slight salty-briny. Some of the samples had a small amount of dark flesh with a hard-to-define character related to gamey and fish oil. The aftertaste is sour, fresh fish, and sweet, with a persistent mouth-drying. Metallic and stale fish notes can be found with this fish also (48, 49, 37).

### 14. Salmon

Flavor profiles of fish aroma and intensity differ among species. For fresh salmon, a fish oil characteristic is common. Also, bitter, metallic, nutty/buttery, and sour characteristics are often observed. Homogenates of salmon stored for 26 weeks at  $-60^{\circ}$ C and  $-13^{\circ}$ C and evaluated using aroma extract dilution analysis (AEDA) and gas chromatography-olfactometry (GC-O) gave some of the odor descriptors such as sweet, buttery, vegetablelike, green, cabbagelike, mushroomlike, citruslike, and cucumberlike. Green (Z-3-hexenal) and fatty green (Z,Z-3,6-nonadienal) are responsible for flavor defects in boiled salmon when stored frozen for a long period. The levels of these two compounds are higher in salmon because of its higher content of n-3 unsaturated fatty acids (53).

Components of low volatility can be the key compounds causing off-flavor. Formation of less volatile oxidation products could also explain why the volatile components identified in the stored salmon did not show significant time—temperature interaction effects nor did they correlate to the sensory attributes.

The most noticeable sensory change during frozen storage of salmon is an increase in intensity of train oil taste, bitterness, and metal taste. This change is produced by compounds of low volatility but is most of all due to free fatty acids. Palmitoleic acid and linoleic acid, eicosapentaenoic acid, and docosahexanoic acid have high intensity of train

oil, bitter, and metallic taste (66). Frozen salmon (*Salmo salar*) become rancid after a few months of frozen storage. Also, fat content of fish diet has a direct relation in some sensory attributes such as color (redness) and the taste of salmon. Fish fed with a 17% fat diet have shown differences in color (less red) and taste (less strong). Frozen storage temperature also influences color, hedonic consistency, hardness, and juiciness (67).

Rancidity in fatty fish such as salmon is not well characterized and has been described as fish oil taste (67, 68) and as fatty and train oily odors (53). The rancid off-flavor in salmon is caused by the formation of volatile oxidation products such as aldehydes and ketones (53). Some of these volatile compounds have very intense odors and flavors and are, even in small concentrations, able to affect the sensory quality. The rancid off-flavor of salmon is mainly caused by an increase in (E,Z)-2,6- nonadienal with a cucumber odor, (Z)-3-hexanal with a green odor, and (Z,Z)-3,6-nonadienal with a fatty odor (53).

The odor of fresh raw salmon is characterized as cucumberlike with weak sweet, sourish, and fish oil notes. For fresh and cooked salmon, the same descriptors were used, but a boiled potato odor has been described as the most pronounced attribute instead of cucumber.

Panelists normally recognize sensory changes of frozen salmon. For train oil, metal, and bitter taste, significant time–temperature relations have been determined. The intensity of these attributes increased during storage at  $-10^{\circ}$  and  $-20^{\circ}$ C. Changes in texture are also noticed at  $-10^{\circ}$  and  $-20^{\circ}$ C. Fresh cooked salmon is described as flaky, firm, and juicy. After storage, salmon changed to firmer, less juicy, and more fibrous (69, 70).

# 15. Shrimp

Texture, color, and flavor of shrimp are influenced by frozen storage. Quality of frozen-stored shell-on and shell-off prawns at  $-29^{\circ}$ C depends on the thawing method and the frozen storage time. Muscle proteins of freshwater prawn tails are susceptible to freezing—thawing processes, particularly during the first month of frozen storage. Protein destabilization is independent of the freezing method (blast versus still) and the presence or absence of shell, but is affected by thawing rate (71). Quick frozen prawns (*Machrobrachium rosenbergii*) stored at  $-18^{\circ}$ C for up to 225 days and tasted after 1 day of freezing were rated acceptable. At 133 and 225 days, prawns had acceptable texture but thawing time must be increased from 4 hours, for the 133 days old, to 24 hours for the 225 days old. Moreover, prawns should be cooked unthawed or after thawing for no more than 4–8 hours to retain its texture (72).

Shrimp are frozen shortly after catch and kept frozen until they reach the consumer. Shrimp kept in frozen storage can develop white spots in the shell, which definitely influence their appearance. During continued frozen storage the spots increase in size, so the quality decreases. White spots are usually developed after 25 days of frozen storage, but this is a result of the time a shrimp takes to pass through the production process. More important is the storage temperature. Alternating storage at  $-25^{\circ}$ C and  $-29^{\circ}$ C results in increased white spot formation compared to constant storage at  $-29^{\circ}$ C (73).

Sensory variables for cooked, peeled, and individually frozen shrimp (*Pandalus borealis*) include evaluation of natural red color, yellowish discolor, dehydration, shininess, smoothness, natural shape, total odor intensity, fresh odor, stale odor, fishy, ammoniacal odor, urinelike odor, sour odor, old seaweed odor, sulfide odor, rancid odor, cardboard odor, mudlike odor, total flavor intensity, fresh, sweet flavor, stale, fishy, sour, metallic, acrid, cardboard, stockfishlike flavor, rancid, mudlike, salty, and aftertaste (74).

# 16. Squid

Studies indicate that squid has excellent freezing characteristics. Glazing or packing of the frozen blocks is essential in order to prevent desiccation during frozen storage. Quality tests indicate that frozen squid at  $-18^{\circ}$ C or lower can be kept for one year in storage. The sensory characteristics to be assessed in a sensorial examination are appearance, texture, and odor of raw product, and texture, odor, and flavor of cooked product. Freshness of cooked squid can be expressed in a 10-point flavor score scale that includes fresh (sweet, meaty), slight loss of freshness (creamy, sweet, meaty, metallic), slightly sweet (slightly meaty, creamy, milky), no sweetness, slightly sour, sour (musty, cabbage), slightly bitter (overripe cheese, oily, slight sulphide), bitter (sulphide), and strongly bitter. Texture can be grouped into three categories: (a) normal (firm and rubbery); (b) borderline (mushy, slightly sticky, or very firm and rubbery); (c) unacceptable (extremely mushy and sticky or extremely firm and rubbery) (75).

Squid is one of the most highly potential sources of fish protein. Frozen foods and surimi-based products using squid as raw material have become a trend in recent years. The mantle muscle of squid has a specific toughness that is maintained after a second freezing and thawing process. The mantle toughness (hardness) of fresh Argentinian squid (*Illex argentinus*), neritic squid (*Loligo edulis*), and cuttlefish (*Sepia pharaonis*) increases after frozen storage after 120 days. The protein pattern of Argentinian squid mantle refrozen and thawed does not change. Histological samples show that muscle fibers are injured and aggregated over time as these changes are the ones responsible for the toughening of mantle (76).

## 17. Striped Bass

Striped bass has a total intensity of aroma above moderate and a total intensity of flavor at moderate. It is a moderately strong flavored fish with a fresh, gamey, fish oil character. The aroma and flavor amplitudes are moderate, with character notes blended to give a full aroma and flavor. The aroma has an early impact of fresh and gamey fish at slight to moderate, slight sweet, sour and briny, and below slight fish oil. The flavor also has an early impact of fresh, gamey fish, and sour notes at slight to moderate, sweet, salty-briny, and mouth-drying at slight, and fish oil at below slight. The sour flavor note is stronger than in most fish. Particularly in flavor, the gamey and fish oil vary, being more intense in the dark flesh. The aftertaste is sweet, sour, and fresh fish, with some gamey fish character (48, 49, 37).

## 18. Swordfish

Swordfish has a moderately low total intensity aroma and flavor, with a sweet, fresh fish, shell fish character. The aroma and flavor amplitudes are moderate, complex, and tightly blended, and the fish has full flavor, with complex first notes, sour (sharp), and mouth-filling characteristics. The aroma is just below moderate fresh fish, slight to moderate shellfish, slight sweet and briny, below slight sour, and very slight nutty-buttery. The flavor is just below moderate fresh fish, slight sweet, shellfish, mouth-drying, and mouth-filling, slight to moderate sour (sharp, citric), very slight canned salmon, salty-briny, and fish oil. The fish oil appears late in the flavor. In aroma and flavor, the fresh fish, shellfish, and nutty-buttery seem rich, closely related, and to have a salmon or tuna character. The canned salmon flavor note is also related and hard to define. The sour (citric) flavor is almost lemon and is stronger than in most of the fish tested. The aftertaste is sour, fresh fish, sweet, and shellfish (48, 49, 37).

### 19. Tilefish

Tilefish has a moderately low total intensity aroma and flavor, with a sweet, fresh fish, shellfish character. The aroma and flavor amplitudes are moderate, as this fish is rich and full, complex, and blended. The aroma is slight to moderate fresh fish, below slight sweet and sour (pungent), and slight shellfish and briny. The sweet, fresh fish, and shellfish notes seem closely related and associated with a rich character like butter, lobster, or scallop. The aftertaste is fresh fish and sweet (48, 49, 37).

### 20. Trout

Frozen fillets of rainbow trout (*Oncorhynchus mykiss*) stored at  $-18^{\circ}$ C, wrapped with either polyethylene or polyethylene/polyamide (vacuum), and subjected to a light/dark environment have shown that packaging materials have a strong effect on the development of rancid flavor. Also, the fillets stored in darkness generally received better appearance scores than the ones stored in light (77).

### 21. Tuna

White tuna (*Thunnus alalunga*) possess white color, firm texture, flavorful flesh, and high nutritional content. To extend production, frozen albacore tuna is used as a raw material in the canning industry. Measurement of sensory, chemical, and physical changes has shown that deterioration of fish quality continues during frozen storage. Quality tests include visual appearance evaluation based on general external appearance, eyes, gills, consistency, and ventral cavity. These parameters are used to classify albacore tuna in four quality categories (highest quality, good quality, fair quality, and rejectable). Frozen tuna quality is good after 12 months of frozen storage. However, after 6 months some specimens developed a slight rancid odor (78).

### 22. Weakfish

Weakfish has a moderate total intensity aroma and flavor, with a weak fresh fish, gamey, briny (seaweed) character, and a late, low level fish oil flavor. The aroma and flavor amplitudes are below moderate, as the character notes are not fully blended. The aroma is slight to moderate briny (stronger than in most fish, with a seaweed character), slight fresh fish, gamey and sour, and very slight sweet. The flavor is slight to moderate fresh fish, slight sweet and sour, below slight gamey, and salty-briny, above slight mouth-drying, and very slight fish oil. Gamey and fish oil are more intense in the dark flesh. The aftertaste is mouth-drying, fresh, gamey fish, with sweet, and sour notes. Compared to other fish, this aftertaste is stronger and more persistent. The character notes of individual samples varied more than in most fish, especially in the levels of gamey and fish oil notes, which depended largely on the amount of dark flesh present (48, 49, 37).

#### 23. Minced Fish

Turbot (Atheresthes stomias), grey cod (Gadus macrocephalus), dogfish (Squalus acanthias), shortspine thornyhead (Sebastolobus alascanus), pollock (Theragra chalcogramma), redbanded rockfish (Sebaster babcocki), and ocean perch (Sebaster alutus) have been evaluated to produce frozen minced fish flesh. Significant differences were found after one month of storage in odor and taste quality. Pollock, pollock/red-banded blend, and ocean perch were given relatively high odor and taste scores. After 3 months, no significant

Table 4 Aroma, Appearance, Texture, and Flavor Attributes Measured in Selected Fish

	Sensory attributes					
Product	Aroma	Appearance	Texture	Flavor		
Catfish (Clarias gariepinus) (50)	Earthy fresh Rancid (oil) Off-aroma	Color Juiciness Flakiness Sponginess Stickiness	Juiciness Mushy/soft Gelatinous Sponginess Stickiness	Earthy fresh Sweet Salty Sour Earthy muddy Bitter Rancid Off-flavor		
North Atlantic Fish (48)	Briny Sweet Fresh fish Old fish Stale fish Sour Shellfish Gamey fish Fish oil Earthy Nutty-buttery Musty Scorched			Aftertaste Salt-briny Sour (Sharp) Sweet Fresh fish Old fish Stale fish Shellfish Gamey fish Fish oil Earthy Nutty-buttery Canned salmon Bitter Metallic Mouth drying		
Salmon (Salmo salar) (69)	Boiled potato Train odor	Color	Firmness Juiciness Fibrousness	Mouth filling Earthy taste Fish oil Train oil Bitter Metallic		
Cod (Gadus morhua) (61)		Color	Firmness Flakiness Resilience Juiciness Cohesiveness Fibrousness Roughness	Strength Fresh fish Saltiness Sweetness Stale Bitterness		

differences in odor quality were found among the different fish species. Differences were found in taste quality, turbot and ocean perch having the highest scores. After 5 months, there were no differences in odor quality, but significant differences were found for taste quality. After this point pollock was scored unpalatable. Texture of the flesh of the different species can be grouped into three categories: relatively firm, intermediate in firmness, and relatively soft. Color of the minced flesh for all species changes only in the lightness of hue (79).

Atlantic cod (*Gadus morhua*) frame mince without kidney tissue, responsible for "chemical" and "petroleum" type flavors, was stored at  $-14^{\circ}$  or  $-40^{\circ}$ C. Aroma, juiciness, texture, and flavor of the cooked samples was evaluated. Instron hardness scores were reduced during storage observing a soft, mushy texture. Color is one of the most important factors for mince quality. Panelist scored fillet mince samples juicy with a soft and moderate texture. No off-odors or off-flavors were detected. Frame mince samples were also juicy but more fibrous and firm than fillet mince (80).

Raw, thawed cod mince texture is commonly evaluated for springiness (0 = completely plastic, 10 = very springy) and stickiness (0 = breaks down, 10 = very cohesive). The frozen deterioration of cod mince is reflected in an increase of cohesiveness (81). Texture scores in cod frame mince stored at  $-7^{\circ}$ ,  $-14^{\circ}$ , and  $-40^{\circ}$ C for springiness and cohesiveness are higher for mince stored at  $-7^{\circ}$  and  $-14^{\circ}$ C. Changes during frozen storage change sensorial properties of the mince (82). Quality changes of fish muscle are normally due to autolytic chemical reactions, microbial proliferation, and physical property alterations. Frozen storage of catfish mince at  $-20^{\circ}$ C can increase storage life to at least 3 months (83). Washed and unwashed catfish mince do not change color during frozen storage at  $-20^{\circ}$ C (84). Washing of catfish mince using sodium citrate, sodium erythorbate, sodium citrate plus sodium erythorbate, and polyphosphate reduce lipids and increase Hunter "L" values (85). Table 4 provides common sensory attributes of selected fish species.

# E. Fruit and Vegetables

The most common problems in frozen fruits and vegetables are reactions that cause changes in flavor, color, texture, or nutritional value. A major result of freezing fruits and vegetables is usually a loss of tissue firmness. Freezing causes disruption of the membranes of cells, and thus excessive softness (86).

Fast freezing rates produce a large number of small ice crystals that cause less damage. Freezing large-size foods involves problems due to thermal gradients. With the high-pressure-shift method, samples are cooled under pressure ( $200\,\mathrm{MPa}$ ) to  $-20^\circ\mathrm{C}$  without ice formation, and then pressure is released to atmospheric pressure ( $0.1\,\mathrm{MPa}$ ). This technique leads to uniform and rapid ice nucleation throughout the volume of the specimen. This method maintains the original tissue structure of the sample (87).

Blanching is often used to inactivate undesirable enzymes that cause unfavorable effects on the quality of frozen vegetables and some fruits. The use of peroxidase as an indicator of the blanching process may result in the loss of color, flavor, texture, and nutrients of vegetables and fruits. Off-flavors from the deteriorated vegetables or fruits can result from the oxidation products created by the action of free fatty acids and/or peroxide degrading enzymes (88).

#### 1. Fruits

Fruits as a class are the most difficult of all products to freeze without causing a radical change in their appearance, texture, flavor, and color. Of all the common fruits, the cranberry is about the only one that can be frozen without special treatment and yet retain its flavor, color, and appearance. All other fruits require packing in sugar or syrup, or some other treatment. Without it, most blackberries turn brown and become sour and flavorless (89). Many of the tropical fruits are even more difficult to freeze. Bananas turn

black during thawing. Whole oranges may break open, and upon thawing, become very flabby and bitter and lose their characteristic flavor (89).

Raspberries, frozen without sugar or syrup, become very soft and lose their characteristic flavor. Most plums become so sour that they are almost sharp and also become very flabby. Blueberries get very soft and lose much of their flavor and aroma. Unripe fruit, which is still hard, does not yield a product of desirable texture, flavor, aroma, and color. Immature fruit when frozen and thawed is usually very sour and often very bitter; furthermore, color and aroma are usually lacking. Moreover, browning and discoloration are more pronounced, and as a rule the texture is not good (89). Frozen fruits allowed to stand after thawing are not as palatable as when served while still slightly frosted. They tend to collapse with resultant poor texture. Unless treated to prevent change, they discolor (90). Thus the nutrients, the desirable juices, the sanitary quality, and the firm texture of fresh frozen foodstuffs may disappear with varying rancidity once the produce is left in the thawed condition.

Tree-ripened or vine-ripened fruit usually yields a better frozen product, as it is generally considered to have better color and flavor than fruit that has been picked while still immature and ripened in storage (89). On the other hand, overripe fruit bruises easily and deteriorates rapidly even when handled carefully and can develop undesirable off-flavors in the frozen product (89).

Most of the common varieties of blackberries and many of the dewberries turn brown and become rather sour; consequently, most varieties are not suitable for freezing in small packages for use as desserts (89). The English Morello variety is preferred for certain purposes because of its more pronounced flavor. It is much darker in color than the Montmorency and is considered less desirable for freezing because of this characteristic (89). Some authorities recommend the Early Richmond variety for freezing, but although this cherry has a desirable bright red color it is likely to be soft and lacking in flavor (89). The Royal Duke and May Duke varieties possess a more pleasing cherry flavor than the standard red sour cherry, the Montmorency. They are more meaty and consequently do not collapse on freezing and thawing to the same extent as does the Montmorency (89). Most varieties of sweet cherries brown (oxidize) badly during freezing, storage, and thawing. A marked change in flavor occurs simultaneously. Sweet cherries are mild in flavor; freezing and thawing cause some loss of flavor, so frozen sweet cherries are likely to be lacking in flavor (89).

The Mission variety of figs is considered best for freezing because of pronounced flavor, good texture, and attractive color. The Kadota and Adriatic varieties remain unchanged in appearance but not so in the case of flavor. The Calimyrna variety is inferior because of the development of off-flavors in the skin and therefore unsatisfactory unless peeled before frozen. Brown Turkey discolors badly and has inferior flavor after being frozen (89).

New York State grapes retain flavor and other characteristics during freezing and subsequent storage (89). Muscat of Alexandria retains flavor and appearance best after being frozen. The Thompson Seedless had a flavor loss, but remain pleasantly sweet after frozen storage.

Pears become soft and watery during freezing and thawing. They also lose much of their flavor. Because of these undesirable changes, pears are not recommended for freezing (89).

Black raspberries possess a delectable flavor and are not altered appreciably by freezing. Unfortunately, black raspberries have a tendency to be seedy; further, the seeds are much more noticeable and objectionable after freezing and thawing (89).

In order to be desirable for freezing preservation, a strawberry should have a pleasing potent flavor and acidity that should be retained during freezing and thawing; it should be a uniformly deep, bright red and should retain this color during a long period of freezing storage. The berries should be of uniformly good size and of firm texture.

Color is one of the most important attributes of food, both for its aesthetic value and for quality judgment. In strawberries, two anthocyanin pigments mainly determine the red color: pelagornidon (Pgd)-3-glucoside and cyanidin-3-glucoside in a ratio 20:1. These pigments are not very stable chemically and may change easily if not properly protected (89). Freezing is one of the most important unit operations to retain fruit quality during long-term storage. Raspberry fruits (*Rubus idaeus* L.) frozen at  $-80^{\circ}$ C and stored at  $-20^{\circ}$ C retain sensorial (color and aroma) and nutritional qualities after 12 months (91). Freezing does not change the volatile components that contribute to the aroma of raspberry (-pinene, citral, -pinene, phellandrene, linalool, -ionone, caryophyllene, and -ionone). However, freezing causes some cellular disruption that increases the release of caryophyllene. The volatile constituents of raspberry aroma remain after one year of frozen storage (92).

Papayas have unusually high amounts of bound water. This bound water has a high affinity for polysaccharides and proteins and remains unfrozen even at  $-20^{\circ}$ C. Frozen storage develops off-flavors and color modification after prolonged storage as a consequence of enzyme action. When papaya slices are cryogenically frozen at  $-80^{\circ}$ C and stored at  $-24^{\circ}$ C for 12 months, proteins are denatured and the activity of soluble peroxidases increase as a result of mechanical damage produced by the growth of the ice crystal and leading to color and texture modification (93).

Storage of custard apple (Annona squamosa L.) has limitations, since it is perishable, and cold storage is not promising owing to the development of brown color. The custard apple pulp is frozen at  $-25^{\circ}$ C and stored at  $-18^{\circ}$ C. The sensory attributes were evaluated using a 10-point hedonic scale, and the conclusion was formed that frozen pulp quality is affected by freezing as compared to the fresh pulp (94).

The flavor of most fruit juices can be preserved better by freezing than by any other method of preservation. Despite this fact, the freezing of fruit juice has not developed nearly as extensively as the other food freezing industries. The two principal reasons for this are (a) properly prepared flash pasteurized juices are nearly as good, and may be kept at ordinary room temperatures without appreciable change of flavor; and (b) the thawing of juices requires much time, and if it is not carefully carried out, the thawed juice may be inferior in quality to a good grade of pasteurized juice.

If berries are pressed without heating or freezing, the yield of juice is low, and the juice obtained is pale in color and very mild in flavor; it oxidizes very quickly owing to oxidative enzyme action (89).

Freezing and thawing of berries prior to pressing is desirable, as it extracts much color and relatively little tannin (89). When Montmorency or English Morello cherries are pitted, much juice leaks from the fruit. This juice is usually saved, and some of it is preserved by freezing. Although this juice is of fine flavor, it is very pale in color and so is not considered to be of high quality (89).

In the case of orange juice, slow-frozen juice is likely to have an "oxidized" flavor, whereas properly handled quick-frozen juice should not have this off-flavor.

The slush-freezing operation requires only 6 minutes, so the system is very effective in rapidly reducing the temperature of the juice to the point where changes in flavor take place very slowly.

Concentration of fruit juices involves sublimation; it is possible to dehydrate fruit juice to a dry powder without materially changing its flavor, although some of the more

volatile components are lost. The process not only produces less change in flavor than any other method of dehydration but also yields a product that rehydrates almost instantaneously.

If the original flavor of the juice is to be retained unimpaired, it is important not only to thaw the juice quickly but also to keep the juice below approximately 50°F.

The anthocyanin contents of certain juices, i.e., strawberry, significantly decreases during frozen storage, and the addition of sugars significantly improves the pigment retention (89).

# 2. Vegetables

It is more difficult to produce frozen vegetables with textural quality than to produce quality frozen fish and meat. Textural qualities of fruits and vegetables lessen upon freezing and thawing, resulting in excessive softening (95, 86, 96,98). This is the prime defect of frozen fruit and vegetable products.

There is no single key enzyme that is responsible for all the vegetable quality changes during frozen storage. Quality changes include off-flavor development (lipoxygenase, lipase, protease), textural changes (pectinase, cellulose), and color changes (polyphenol oxidase, chlorophyllase, peroxidase, lipoxygenase) (99). In general, if the blanching treatment is not sufficient to inactivate catalase and peroxidase, frozen vegetables will develop off-flavors. The development of haylike flavors, bitterness, and odors in unblanched frozen vegetables may be due to amino-aldehyde reactions (100).

The effect of low-temperature long-time blanching (60°C for 2 hours or 74 to 79°C for 20 to 3 minutes) on improvement of textures of frozen vegetables was observed in several studies on different foodstuffs preserved by different methods, such as canned vegetables (101, 102) and frozen carrots (103, 100, 104).

Processing conditions like blanching and freezing result in substantial dissolution, depolymerization, and apparent destruction of cell wall pectins. As a result, pectins are broken down into smaller polymers, lose interpolymer associations, are released from the primary cell walls and middle lamellae, or remain loosely bound to the walls by relatively sensitive bonds (101–103, 100, 104).

### 3. Mushrooms

Since most vegetables and fruits consist of many kinds of enzymes, enzymatic reactions such as the oxidation reaction, the browning reaction, and discoloration present a problem to the frozen vegetable industry. In order to keep better quality, including color, flavor, aroma, taste, texture, and appearance of the frozen vegetables, many chemical additives are occasionally used for this purpose. Because mushrooms are one of the easily browning vegetables caused by enzymatic and chemical oxidations, there are many kinds of enzyme inhibitors or antioxidants recommended to prevent those quality lowering. While numerous factors are involved in quality loss in frozen mushrooms, the most detrimental is the enzymatic browning reaction (105).

Problems may occur during freezing and storage of frozen mushrooms, e.g., loss of weight, decrease in nutritive value, tendency of product to stick together, and sometimes development of undesired color and off-flavor. Shelf life of fresh mushrooms (*Agaricus bisporus*) is limited to 1 to 3 days at room temperature, 5 to 7 days at 0 to 2°C. Washing, freezing, and 1 day storage of mushrooms contributed to the loss of "L" value. No changes have been reported in whiteness of frozen mushrooms after 90 days of storage. However, washing with water increases browning of frozen mushrooms. Immersion in

boiling water increases toughness as the storage time of frozen mushrooms increased more than 14 days. The blanching process itself reduces the initial mushroom whiteness (106), and blanched frozen mushrooms show a remarkable toughness after thawing and cooking. Frozen whole mushrooms blanched for 5 minutes have a tougher texture than those blanched for 1 or 2 minutes.

Mushrooms immersed in boiling water before freezing are quite acceptable for the consumer for all time storage even though there is typically a loss of whiteness as compared with mushrooms washed in water containing sodium metabisulfite. Dipping mushrooms in 0.1% or 0.5% sodium metabisulfite solution for 5 minutes after blanching has resulted in the best color and other quality characteristics after freezing (107) of mushrooms in boiling water, whereas before freezing was harmful to their texture. Appearance and color of unblanched frozen mushrooms were most affected by washing them in sodium metabisulfite solution before freezing. Storage time of frozen mushrooms results in lower discoloration of mushrooms washed in water containing sodium metabisulfite or immersed in boiling water for 20 seconds than mushrooms washed in water only. Their texture (shear-press reading) was higher. Immersion in boiling water containing sodium metabisulfite had a dual effect: a marked loss of whiteness and a significant decrease of SO<sub>2</sub> content. Also, the sodium metabisulfite—treated mushrooms tend to show a marked toughness during storage.

Chemical dipping methods were effective in maintaining desirable color of frozen whole and sliced mushrooms stored for 3 months (108). Excellent results in stabilization of the color of frozen mushrooms by inhibiting undesired discoloration have been obtained after washing fresh mushrooms in sodium metabisulfite solutions (109). The better quality of frozen mushrooms treated with sodium bisulfite solution was obtained before and after blanching. EDTA and NaPO<sub>3</sub> do not improve the color or other qualities such as dripping loss, texture, and sensory evaluation scores.

Sodium bisulfite and sodium diethyldithiocarbanate after blanching has a higher ascorbic acid retention and less dripping loss, but its organoleptic evaluation scores were poor. Since sulfites are essential additives with important effects on the whiteness stability of frozen mushrooms, determination of the minimum amount of SO<sub>2</sub> required to maintain optimum frozen mushroom quality seems to be necessary.

Loss of frozen mushroom whiteness after 1 d has been compared with fresh ones varying an average of 6% to about 17%. No significant change in whiteness of frozen mushrooms during 90 days of storage was detected when short-time dipping in boiling water was applied. Storage time of frozen mushrooms results in lower discoloration (110). Most mushrooms are frozen commercially by the air blast or plate freezing methods. However, after freezing some undesirable color and off-flavor may occur. The frozen mushrooms made by individual quick freezing process were better in quality, firmer in texture, and lower dripping loss than those made by air blast freezing process.

The effects of processing vary with the chemical, physical, and sanitary conditions and sensory quality of the frozen mushrooms. IQF mushrooms show better color and higher sensory scores. After 120 days of frozen storage, color and texture of mushrooms changed (107). Frozen whole mushrooms blanched for 5 minutes had markedly tougher texture than mushrooms blanched for 1 or 2 minutes. As the storage time of frozen mushrooms increased from 3 to 6 months, shear press values increased very significantly. The texture of Freon frozen and plate frozen mushrooms was similar after 3 months storage. Blanching time did not affect texture of frozen sliced mushrooms significantly, but storage time did.

# 4. Leafy Vegetables

Texture is important in frozen leafy vegetables. After freezing and thawing, firmness and crispness decrease and rupture strain increases. The optimum freezing rate of Chinese cabbage has been established at 5°C/min because of its high moisture, low sugars, and thin cell walls; therefore freezing effects are more notable. Chinese cabbage frozen at 100 MPa and at 700 MPa shows an increase in rupture strain. However, texture of samples frozen at 200 MPa, 340 MPa, and 400 MPa keep intact (111).

The green color of organically grown spinach (*Spinacia oleracea* L.), evaluated by sensory evaluation and Hunter "L" value after 6 months of storage at -24°C, shows that color and sensory attributes of spinach depend on the cultivar, and these differences should be considered during freezing (112). Lipid-acyl hydrolases (LAHases) are responsible in lipid degradation during storage of vegetables. LAHases are found in spinach that naturally contains large amounts of galactolipids and phospholipids, substrates for LAHases. Formation of volatiles is the result of oxidation of lipids in vegetables. The enzymes involved in this process are lipases, phospholipid-degrading enzymes, galactolipid-degrading enzymes, and lipoxygenase. In spinach leaves, unsaturated fatty acids are present in large portions and are oxidized to peroxides. The resulting peroxides are decomposed to aldehydes, ketones, and alcohols, causing off-flavors (88).

## 5. Asparagus

Studies have shown no significant difference in texture, flavor, or general acceptability in asparagus owing to blanching treatment (113). Blanching of green asparagus (Asparagus officinalis L.) before freezing is necessary to inactivate enzymes that cause quality changes during frozen storage. One of the changes is the formation of an aldehyde such as hexanal, which is described as a green aroma and is a flavor compound formed during autooxidation of linoleic acid. There is no difference in texture of asparagus frozen using cryogenic and blast freezing, but there are differences in texture when asparagus are kept at -30 or -70°C. During storage, shear force values slightly increased (114). Blanched asparagus stored at -18°C for 4 weeks and evaluated for appearance, color, texture, natural flavor, off-flavor, and general acceptability showed that unblanched asparagus scored the highest in appearance, while microwaved and unblanched asparagus had the highest color scores. Flavor of asparagus changed regardless of the blanching treatment. After frozen storage, unblanched asparagus stems were significant darker than blanched stems. Blanching had no effect on lightness of asparagus stems or tips. Asparagus were greener after blanching and less green after frozen storage, also they were yellower after frozen storage (113).

#### 6. Carrots

Freezing destroys the cytoplasmic structure, producing loss of turgor, weakness of the cell wall, and some degree of cell separation. All these changes affect texture of the product. The freezing temperature is the most critical factor; it changes the cell structure of carrots. The rate and type of freezing are the critical factors in tissue damage. Firmness of frozen sliced carrots decreases by about 50% as compared to raw samples (115). Quick freezing results in better texture than slow freezing in carrots (100). The effect of freezing rate on firmness of raw carrots is greater than that on firmness of blanched carrots. The final temperature of freezing does not affect firmness of thawed carrots. As the freezing rate

from  $0^{\circ}$ C to  $-10^{\circ}$ C was quick, firmness of thawed carrots was maintained. The amount of drip increased as freezing rate decreased. Effect of freezing rate on firmness of blanched carrots has been shown to be the same as that of raw carrots. Consequently, quick freezing  $(-5^{\circ}\text{C/min})$  is best for acceptable texture of frozen carrots (100). Tamura (116) found that when carrots were preheated for 30 minutes at  $60^{\circ}$ C, frozen at  $-30^{\circ}$ C, thawed in a refrigerator, and then cooked, softening of carrots was greatly diminished, but cell damage was observed. Carrots frozen at  $-30^{\circ}$ C had great amounts of cell separation; the optimum rate of freezing for carrots has been determined to be  $-5^{\circ}$ C/min (117). Low-temperature blanched carrots frozen at  $-5^{\circ}$ C/min using a program freezer should have minimization of softening. Preheating increases firmness and rupture strain of carrots, while freezing and thawing decreases firmness and increases rupture strain. Firmness of carrots decreases when frozen and thawed, except for raw carrots frozen in a program freezer and thawed. Raw and preheated disks frozen quickly  $(-5^{\circ}\text{C/min})$  using a program freezer are typically firmer than those frozen at  $-35^{\circ}$ C using a conventional freezer. Differences in firmness of blanched disks as compared to unblanched have not been noted. Quick freezing results in better texture (firmness and rupture strain) than slow freezing, and also improves the texture of frozen carrots. Texture of frozen carrots differs from that of boiled carrots (100). Blanching, high-temperature-short-time (HTST), and rapid freezing at  $-4.5^{\circ}$ C/min is recommended as optimum thermal processing and conditions for improvement of textural quality in frozen carrots (118).

Also, freezing causes extensive degradation of cell wall pectins in carrot tissue. This is evident by the loss of firmness. Freezing at rates of  $-4.5^{\circ}$ C/min causes less softening than slow rates ( $-0.19^{\circ}$ C/min). Slow rates causes loss of pectin material and structural damage from growing ice crystals (118).

Before freezing, carrots are blanched to inactivate enzymes that cause flavor deterioration during frozen storage. Unblanched carrots develop an off-flavor owing to fatty acids released by esterases. The off-flavor is characterized as "stearin," "paraffin," and "soap." Loss of textural quality during blanching, freezing, and thawing includes dehydration, damage, drip loss, tissue fractures, and mechanical damage from ice crystals during freezing. Steam-blanched carrots subjected to blast or cryogenic freezing and stored at  $-24^{\circ}$ C for 5 months showed differences in textural quality between blast and cryogenic freezing. Decreasing the temperature from  $-30^{\circ}$  to  $-70^{\circ}$ C resulted in increased toughness, but no changes are observed over time (119).

Studies have shown that carrots immersed in liquid nitrogen (LN<sub>2</sub>) cracked and became unacceptable in appearance, but those frozen at  $-5^{\circ}$ C/min or  $-2^{\circ}$ C/min using a programmed freezer (final temperature  $-30^{\circ}$ C) had a better texture than those frozen in a conventional freezer at  $-80^{\circ}$ C,  $-30^{\circ}$ C, or  $-20^{\circ}$ C (117).

### 7. Broccoli

Broccoli (*Brassica oleracea*) is a highly perishable fresh vegetable that, at ambient temperatures, will yellow and become unmarketable in 1 to 3 days. About 37.8% in florets and 61.1% losses of chlorophylls a and b in stems occurred during freezing storage at  $-20^{\circ}$ C (120). This loss of green color is a major limiting factor in the shelf life of broccoli. Boiling water blanched broccoli stored at  $-18^{\circ}$ C for 4 weeks has excellent sensory scores for visual appearance and color (121). Blanching has a significant effect on yellow color intensity (b\* value, Hunter). Immediately after blanching, stems and florets are more yellow than unblanched samples. These differences remain after frozen storage for florets but not for stems. All blanching treatments increase chroma (color saturation) of broccoli

florets and stems compared to unblanched broccoli (122, 121). To avoid the development of dimethyl sulfide (off-aroma) in frozen broccoli, florets must be blanched for 90 seconds before freezing (123). After frozen storage no significant lightness differences due to blanching treatments have been noted in recent findings.

### 8. Green Beans

Brewer et al. (124) reported no difference in either visual color or appearance scores of frozen green beans due to blanching treatment. Brewer et al. (124, 121) found that blanching treatment had significant effects on flavor, off-flavor, and the tenderness and crispness of green beans and broccoli that had been in frozen storage. Brewer et al. (124) reported that blanched green beans were greener (higher hue angle) than unblanched beans; this trend was still evident after frozen storage.

Samples of green beans have been shown to be more yellow (higher Hunter b\* value) after frozen storage than immediately after blanching. Immediately after blanching, blanched asparagus tips were yellower than fresh and blanched tips. These differences were lost after frozen storage. No differences in yellowness of stems existed either immediately after blanching or after frozen storage. Brewer et al. (121) reported similar blanching and freezing effects on yellowness of broccoli florets and stems. Similar color changes have been reported in blanched green beans during frozen storage (125). Sensory characteristics, such as color, texture, and flavor, are affected by blanching. For green beans, appearance, beany aroma, sweetness, green flavor, aftertaste (metallic/astringent), tenderness, crispness, and wetness are the principal sensory attributes to evaluate in frozen products. After four weeks of frozen storage, color values drop by 50%. Blanching treatments have no effect on visual color, appearance, aroma, sweetness, raw starchy flavor, green bean flavor, overcooked flavor, aftertaste, tenderness, crispness, or wetness of frozen green beans (124). After 12 weeks of frozen storage, panelists detect no differences in color, sweetness, aroma, firmness, and presence of off-flavors. Off-flavor is detected in frozen cooked samples that are blanched using microwaves. This off-flavor is described as grassy, bitter, and stored (122).

Bennion (126) stated that two results of chemical changes that may occur during frozen storage of foods are off-odors and off-flavors. Campbell et al. (127) noted that one of the principal reasons for off-flavors is rancidity of the fatty material in unblanched frozen vegetables. Noble and Winter (128) reported that in unblanched frozen green beans, marked off-flavors developed rapidly by the end of four weeks. Off-flavor descriptions for unblanched frozen green beans reported by Moriarty (129) included earthy, sour, and bitter.

Bennion (126) suggested that off-odors in frozen raw or insufficiently blanched vegetables may be attributed to the accumulation of volatile carbonyl compounds during frozen storage. Chow and Watts (130) reported that rancid odors in frozen green beans were related to malonaldehyde values, used as an index of unsaturated fatty acid oxidation. Lipid oxidation as determined by both the thiobarbituric acid test and acetaldehyde formation from anaerobic fermentation contributed to flavor deterioration in frozen green beans.

After frozen storage, steam blanched and boiling water blanched beans had the lowest shear values. Visual color was poorest for microwave blanched. Microwave blanching green beans for 3 minutes in a covered container or bag prior to 4 weeks frozen storage resulted in a product that was not different in retention of color from boiling water

blanched beans; however, these bean samples differ in tenderness and crispness from boiling water blanched beans.

Katsaboxakis and Papanicolaou (125) found that blanching green beans (30–90 seconds) increased negative  $a_{\rm L}$  values and the  $a_{\rm L}/b_{\rm L}$  ratio during frozen storage. Negative Hunter "a" values measure sample greenness. Unblanched green beans have higher odor and flavor scores before storage but are considered organoleptically unacceptable for the same attributes after the first months of frozen storage and at all subsequent storage times. Samples blanched for 30–90 seconds are slightly superior in sensory qualities to those receiving more drastic heat treatment.

Hue angle [(tan<sup>-1</sup> (b/a)] has been shown to be higher for boiling water, steam, and microwave blanched fresh beans. The same general trend for hue angle was still evident after 4 weeks in frozen storage and cooking. Higher hue angles indicate that the sample color was greener.

After 4 weeks in frozen storage and cooking, chroma was greater for steam and microwave blanched beans than for unblanched green beans. Total color values drop by nearly 50% after storage and cooking for most blanching treatments; total color of microwave blanched beans has been shown to drop over 70%. Katsaboxakis and Papanicolaou (125) reported significant color loss (Hunter L value, a/b ratio) owing to green bean blanching time and time in frozen storage.

After frozen storage, blanching treatment had no effect on visual color, appearance, aroma, sweetness, raw starchy flavor, green bean flavor, overcooked/canned flavor, aftertaste, tenderness, crispness, wetness, or degree of liking of cooked green beans. Microwave blanched beans had more aftertaste than boiling water blanched beans (125).

Sensory evaluation after 9 months of frozen storage found no significant difference in green bean off-flavor between short and long blanch times at any storage temperature. Off-color was significantly higher in green beans that were blanched for 63 seconds, however, indicating that in this commodity color may be the limiting factor in frozen storage. In the case of green beans, off-flavor is eliminated by a one min blanch, but off-color prevention may require a longer time (125).

# 9. Peppers

A major problem of jalapeño peppers (*Capsicum annuum*) is the soft texture after exposure to heat and salt brines with a pH less than 3.5. To overcome this problem, jalapenos can be frozen. The maximum firmness of frozen jalapeño pepper is 4.8 times that of unfrozen samples (131).

# 10. Tomatoes

Sensory characteristics of tomatoes include appearance, red color intensity, tomato flavor intensity, texture, natural tomato flavor, off-flavor, and general acceptability. After 6 weeks of frozen storage, boiling water blanched tomatoes have the highest scores for appearance, tomato flavor intensity, and natural tomato flavor. Steam blanched tomatoes have the lowest appearance scores. Unblanched frozen tomatoes are usually mushy and least firm after storage (132). Tomatoes blanched (4 minutes) using conventional boiling water or steam, microwaved in a glass container, or in boilable bags demonstrated that though visual color and sensory attributes were highest for boiling water blanched tomatoes, microwaved blanched tomatoes retained more nutritive value in the finished product (132). Pectin methylesterase (PME) can cause texture deterioration in processed

tomatoes during storage. Blanching, which reduces the loss of product texture, inhibits PME activity as well as the activities of other deteriorative enzymes (132).

Lycopene, the primary pigment in tomatoes, is affected not only by exposure to oxygen from the air but also by heat during processing, which may cause isomerization of trans double bonds in the pigment to cis forms (133). This shift in structure can cause a reduction in color intensity of a variety of food products (134). Preliminary acceptance or rejection of a food is usually influenced by the visual appearance including color and texture (135). Any changes in blanching technique must take into account the alterations in product color that often occur. This reduced, but still substantial, PME activity may be the reason for poor sensory texture of unblanched frozen tomatoes; as PME breaks down pectic substances, tissues lose structural integrity and become soft and mushy (132).

Immediately after blanching, microwave blanched tomatoes have been shown to be lightest (Hunter L value) in color. Boiling water blanched tomatoes are redder (Hunter a value) with a similar trend for yellowness (Hunter b value). However, after frozen storage, steam blanched tomatoes are lighter in color followed by microwave blanched and unblanched tomatoes. Lightness of steam and microwave blanched tomatoes change little during storage. Although total carotene content does not change as vegetables are heated in water, there is a shift in the visual color: orange carrots may become more yellow, and red tomatoes may become more orange-red (135).

Blanched tomatoes have lower hue angles than do fresh tomatoes, indicating that blanched tomatoes are more "true red" than fresh tomatoes. Hue angles decrease after frozen storage.

Chroma increases twofold as a result of the blanching process. After frozen storage, total color drops significantly. After 6 weeks of frozen storage, boiling water blanched tomatoes have the highest scores for appearance, tomato taste intensity, and natural tomato flavor, followed by microwave blanched tomatoes. Brewer et al. (121) found a similar trend for broccoli. Steam blanched tomatoes had the lowest appearance scores. Unblanched tomatoes were most mushy and least firm after storage. No texture differences exist among boiling water and microwave blanched samples.

Begum and Brewer (113) found only moderate acceptability scores for asparagus (3.27–3.48 on a five-point scale) for all blanching treatments and reported that appearance and color appeared to have greater impact on acceptability than did flavor. Unblanched tomatoes had the lowest scores for general acceptability as compared to those blanched by various methods. Stone and Young (122) also reported that, in general, microwave blanched vegetables are less acceptable in terms of texture and flavor than those that are steam or water blanched. Some microwave heated vegetables exhibit toughening and may have grassy or strong off-flavors and aromas (136, 122). These results may be attributed to the amount of water used during blanching, high microwave power level, or extended heating time. However, tomatoes are softer products that are likely to soften even more as pectic substances are solubilized.

Individually quick freezing (IQF) versus blast freezing does not influence the texture of sweet corn or green peas. Also, no changes in color are observed in green asparagus, green beans, or green peas. Asparagus and green beans that are blast frozen show softening, and sweet corn has higher color values (137). For sweet corn on the cob (Zea mays) frozen using a blast freezer, it is recommended to reduce blanching time from the sensory point of view because longer blanching times result in reduced firmness and freshness and also increase cooked flavors (123).

The freezing method does not affect the color of green vegetables but does affect corn. Blast frozen sweet corn has higher blue (less yellow) color than the IQF sweet corn.

In many vegetables the limiting quality attribute during frozen storage is off-flavor development, which is most often catalyzed by lipoxygenase. Williams et al. (138) evaluated the sensory character of blanched vegetable purees to which isolated enzymes had been added and found that lipoxygenase was the enzyme most active in aroma deterioration in English green peas and green beans. Lipoxygenase is widely distributed in vegetables, and evidence is mounting to support its involvement in off-flavor development and color loss.

For corn, green beans, and green peas there is fairly strong evidence that off-flavor development is the limiting factor in frozen storage and that lipoxygenase activity is the primary culprit.

Supersweet corn is popular as a fresh, frozen, or dried product, but blanching often results in the caramelization of these high-sugar varieties and in the formation of an undesirable gray or brown product. Industrial blanch conditions were established based on the requirement for peroxidase inactivation, but off-flavor catalyzed by lipoxygenase is the greatest limitation to storage life.

Sensory results for stored kernel corn indicated that unblanched kernels were significantly less desirable in appearance, texture, flavor, and overall desirability. It would appear that a one min, or possibly shorter, blanch was adequate to ensure the sensory quality of frozen Supersweet kernel corn.

Corn-on-the-cob sensory results show that, although the appearance of both unblanched and 3-minute blanched samples were preferable to those blanched longer, a longer blanch treatment resulted in better flavor and overall desirability after all storage times.

There have been shown to be no significant differences in off-flavor or off-color between the short- and long-time blanched corn after 9 months storage at  $+8^{\circ}$ C,  $0^{\circ}$ F, or  $-8^{\circ}$ F. Table 5 provides common sensory attributes for selected vegetables.

**Table 5** Color, Appearance, Aroma, Flavor, and Texture of Selected Vegetables

	Sensory attributes						
Commodity	Color	Appearance	Aroma	Flavor	Texture		
Green beans (124)	Green	Plump	Grassy/green Beany	Sweet Green bean Overcook Aftertaste	Tenderness Crispness Wetness		
Tomatoes (132)	Intensity	Uniformity of color Wholeness	Aroma	Bitter Natural flavor Sweet Off-flavor	Intact Mushy Firm		
Broccoli (121)	Green	Plump	Grassy/green	Sweet Starchy Broccoli Overcooked Aftertaste	Tenderness Crispness Wetness		

# F. Spices, Herbs, and Seasoning Blends

A spice is any aromatic vegetable substance in whole, broken, or ground form, except for those substances that have been traditionally regarded as foods, such as onions, garlic, and celery; whose significant function in food is seasoning rather than nutritional; that is true to name, and from which no portion of any volatile oil or other flavoring principle has been removed (139).

The shelf life of spices is greatly extended while kept in their whole form, but when ground the shelf life is reduced. The ideal conditions for the storage of spices are to keep them cool, dry, and protected from light. These storage conditions, in addition to airtight packaging, will reduce oxidation. Freezing is a prime choice of storage condition. The freezing of spices has little to no affect on the sensory properties of spices. Freezing is the means by which the gold standard of a spice is typically preserved. The sensory properties of spices and herbs include aroma, appearance, color, flavor, pungency, and in some cases textural aspects. Sensory profiles can be used to define broad parameters, backed by specific and measurable physical parameters such as moisture, particle size, bulk density, color, and, where appropriate, heat units. Several of the physical parameters, such as particle size and solubility, although not primarily responsible for flavor, influence this attribute (140).

The shelf life of a spice, herb, or seasoning blend will depend upon many variables that are typically characteristic of either the raw materials or the packaging, the different characteristics of the spice or herb, or possibly the different types of ingredient (s) used singly or in blended form. Typically, the detrimental properties would be loss of flavor and/or product lumping. Flavor deterioration is a problem when the more volatile ingredients are used, especially spice extracts, oils, and liquid flavors, which have been plated onto a nonabsorbent base, such as salt, dextrose, maltodextrin, or possibly starch. There is virtually a nonexistent barrier by which to hold these volatiles in the compounded blend into which they have been incorporated. Thus even to freeze these types of products would be of little benefit for flavor and aroma retention. From an appearance standpoint, the product could appear slightly faded, but could possibly appear quite normal in color, size, shape, texture, and so on. Rancidity is another concern for flavor and aroma deterioration. In particular, spices that have high oil contents often develop rancidity if not properly stored. Freezing most definitely delays this deleterious property (140).

Flowability is a concern for spices and spice—herb seasoning blends because the loss of moisture in the product causes lumping as well as moisture absorption, causing stickiness with certain spice oils. Free-flow agents are often added to spice blends to assist with this problem. Freezing does not tend to affect these types of spice—herb blends detrimentally. Most seasoning blends typically have a shelf life of 3–6 months if properly stored. However, if frozen, this timeline can be extended to 6 months to 1 year. Many spice houses and distributors store spices, herbs, and seasoning blends up to 2 years without loss of quality or sensory attributes. Cryogenic milling of certain spices avoids the volatilization, oxidation, and enzymatic damage associated with conventional milling techniques. The higher volatile content of these spices (e.g., nutmeg, mace, and cinnamon) provides improved shelf life and flavor perception. Thus using cryogenics these can be extended (140).

## G. Meats

Preservation by some means is absolutely essential for prolonging shelf life and for storage of all fresh meat and most processed meat products. Regardless of the method used, preservation is accomplished by restricting, or in some instances completely inhibiting, microbial activity, as well as enzymatic, chemical, and physical reactions that would otherwise cause deteriorative changes and spoilage. The purpose of freezing is the preservation of the original characteristics of the product for an extended period of time. The main requirement is that the product before freezing is in an optimal condition from both microbiological and chemical standpoints. The quality of frozen meat is affected by conditions during freezing and subsequent frozen storage, and the length of the storage period. Quality changes occurring during storage at temperatures less than  $-10^{\circ}$ C ( $14^{\circ}$ F) are unrelated to bacterial growth or metabolism and are of (bio)chemical origin (e.g., drying and oxidation). In lean muscle at  $-5^{\circ}$ C ( $23^{\circ}$ F), approximately 85% of the water is frozen; at  $-30^{\circ}$ C ( $-22^{\circ}$ F) nearly 100% of the water is frozen. Owing to the presence of solutes, the freezing point of muscle is approximately  $-2^{\circ}$ C ( $28^{\circ}$ F) (141).

The major physical/chemical changes that occur during storage of frozen foods are typically a result of (a) oxidation of lipids, (b) denaturation of proteins, (c) discoloration of product, (d) sublimation of ice, and (e) recrystallization of ice crystals. Chewiness, juiciness, and rancidity are the sensory attributes to be monitored during the storage. Freezing is one of the most effective methods for meat preservation. Low temperature not only protects the product from microbiological spoilage but also slows down the rates of other degradative biochemical reactions. In spite of the many advantages however, freezing causes certain unfavorable changes in meat quality. Ice occupies a greater volume than water, and the exclusion of solutes from ice crystals causes an increase in the ionic strength in unfrozen water. Together these phenomena cause a loss in tissue structure and a partial denaturation of some muscle proteins, in turn reducing protein solubility and gelation capacities (142).

Freezing and frozen storage produce or enable deleterious changes that can reduce meat quality depending on storage time, temperature, freezing rate, and protective packaging used. Secondary breakdown products of lipid oxidation, especially malonaldehyde, have been shown to precipitate on and cross-link muscle proteins, resulting in reduced ability to hold water under physical or thermal condition. Low-molecular-weight aldehydes and ketones are known to have strong odors and flavors at low concentrations, some of which are objectionable to sensory panels (143).

Color may be the most important characteristic affecting consumer decision-making on the acceptability of meat and meat products (143). Such discrimination may be warranted, as meat color is the quality trait most susceptible to alteration by environmental conditions (144). Consumers associate color with good quality (145, 146). Freezing or frozen storage generally does not affect the color of cooked meat (147). In frozen meat, color changes as a result of storage time, temperature, freeze—thaw cycles, and exposure to light (148, 149).

If flesh tissue is frozen rapidly, the cellular fluids remain in their location and freeze as tiny crystals uniformly distributed throughout the tissue. The faster the transition from  $0-5^{\circ}$ C, the less is the translocation of water during freezing (150). Slow freezing results in more thawing drip and less expressible juice than quick freezing (151). Slow freezing causes fluid in the extracellular spaces to freeze first, thus increasing the concentration of solutes and drawing water osmotically from the still-unfrozen cell through the semipermeable cellular membrane. There is therefore extensive translocation of the tissue fluid such that

the fibers appear shrunken and in some cases damaged through rupture of the myofibrillar walls. Drip loss is not only disadvantageous economically but can give rise to an unpleasant appearance. Drip loss is due to denaturation of proteins by the high ionic strength of extracellular fluid.

Drip losses, cooking losses, and shear strength increased with time in frozen storage. Samples with 30% fat had greater drip and cooking losses than did samples with 15% fat. Storage temperatures affected drip and cooking losses but not shear strength. Increased storage time also resulted in loss of color.

Protein damage is a function of time, and the quantity of drip will tend to increase as with time in storage (152). Furthermore, the free moisture is a medium for potential bacterial spoilage and also represents loss of flavor in the meat as nutrients such as vitamins, proteins, and minerals are dissolved in the exudate. Slow freezing rates produce greater damage to the mitochondria in the cells than fast freezing, and consequently there is a greater release of enzymes (153).

Freezing is recognized as an excellent method for the preservation of meat. It results in fewer undesirable changes in qualitative and sensory properties of meat than other methods of preservation. In addition, most of the nutritive value of meat is retained during freezing and through frozen storage. Some loss in nutritive value occurs when watersoluble nutrients are lost in thawing drip, but the amount of drip and nutrients varies with freezing and thawing conditions. Nutrients found in drip include salts, proteins, peptides, amino acids, and water-soluble vitamins. A study by Anon and Calvelo (154) confirmed that drip loss is reduced by shortening the freezing period, although Bechtel reported that protein in the drip was unaffected by the freezing rate. Meat proteins are less affected by freeze concentration; Jalang et al. (155) found that regardless of species, frozen muscle at  $-19^{\circ}$ C provided more expressible (press) fluid than fresh muscle, although pork had more press fluid than beef or lamb. Wagner and Anon (156) explained the effect of freezing rate on denaturing by noting that myofibrillar proteins denature in two stages, with an initial rapid reaction followed by a slower, second stage, with the myosin molecule continuing to denature and leading to a decrease in solubility and viscosity (157). None of the nutrients present in meat were destroyed or rendered indigestible by freezing. The dark color of fresh frozen meat brightens upon exposure to oxygen or air. Thus qualitative properties of frozen meat approximate those of fresh meat (158).

In general, sensory characteristics such as rancid, acid, sour, and bitter odor and flavors, and rubbery texture, increase over time in frozen storage when juiciness decreases. Exclusion of oxygen appears to have the greatest impact on the preservation of sensory characteristics. Based on sensory data, apparently, frozen storage of ground pork should be limited to 26 weeks even with vacuum-packaging if "fresh pork" sensory and physical characteristics are to be maintained (158).

Apart from temperature, the nature of the muscle food product is a decisive factor in determining storage time. Ground product with its greater surface area and potential for oxygen penetration is more sensitive to oxidation (rancidity) than whole pieces of meat. As pork contains more unsaturated fatty acids, it is more susceptible to oxidative rancidity than beef. Undesirable oxidation and dehydration occurring during frozen storage may be limited by proper packaging (159).

The length of time meat is held in refrigerated storage prior to freezing affects the ultimate frozen meat quality. To preserve optimum quality, meat to be frozen must be handled with the same care as refrigerated meat, especially if products are going to be in freezer storage for several months. Some deterioration continues to occur in meat even at freezer storage temperatures. In addition, quality of frozen meat is influenced by freezing

rate, length of freezer storage, and freezer storage conditions, i.e., temperature, humidity, and packaging materials used. Included among changes that may occur during frozen storage are the development of rancidity and discoloration, with the latter change being due to surface dehydration owing to microbial and enzymatic activity. At temperatures below about  $-10^{\circ}$ C, most deterioration caused by microbial and enzymatic activity is essentially curtailed. On the other hand, in meat with relatively high microbial loads or in improperly chilled meat, slow freezing rates may allow considerable microbial growth (158). The main effect of freezing upon microorganisms is a cessation of growth. Some microbial destruction will occur during the freeze/thaw process, but freezing should not be regarded as a reliable means of destroying microorganisms other than parasites (160).

Tempering produces the most rapid increase in myoglobin and lipid oxidation. This is probably the result of both the thawing process and temperature fluctuations. Thawing provides an ideal environment for the formation of new, large ice crystals, and the temperature fluctuations during tempering further enhance ice crystal growth and formation (161). The formation of large ice crystals enhances muscle fiber shrinkage and distortion and increases the solute concentration, which disrupts the integrity of the muscle cells. Such changes permit catalysts of oxidative reactions to come into contact with myoglobin and lipids (162).

Freezing rates affect physical and chemical properties of meat. They may be influenced by the temperature of the freezing medium, the type and movement of the freezing medium, the packaging materials used, and the composition of meat products to be frozen. In the latter case, tissues containing fat have lower thermal capacity than lean tissues and therefore freeze more rapidly (158). A key property for all the components and membranes of muscle foods is their elasticity. The elasticity determines the consequences of freezing and the quality of the resulting thawed material (157).

Undesirable physical and chemical effects occurring in meat during freezing are associated with one or more of the following factors: (a) formation of large ice crystals in extracellular locations, (b) mechanical damage to cellular structures resulting from volume changes, and (c) chemical damage caused by concentration of solutes, such as salts and sugars. Nonvolatile solutes lower the freezing point, and so meat freezes at approximately  $-2^{\circ}$ C to  $-3^{\circ}$ C. The extent of damage to meat tissues attributable to these three factors is influenced by the freezing rate (158).

During slow freezing, temperature of meat products remains near the initial freezing point for an extended time. As a result, a continuous freezing boundary forms and proceeds slowly from exterior to interior. During the process of slow freezing, long periods of crystallization time exist, producing numerous large extracellular masses of ice crystals that are easily lost as drip during thawing. Mechanical damage due to volume changes is more likely to occur during slow freezing because of the expansion associated with the formation of large ice masses, as well as concomitant shrinkage of muscle fibers that have lost water to extracellular pools. Such muscle tissue has distorted structure in frozen form that completely obliterates normal striations (158).

Cryogenic freezing is a rapid freezing method utilizing condensed gases such as liquid nitrogen, nitrous oxide  $(N_2O)$ , and dry ice  $(CO_2)$ . This rapid freezing is primarily the consequence of very low initial temperature. Costs for cryogenic freezing are greater than for conventional freezing. The justification for this method is better achieved quality (163). During cryogenic fast freezing, meat product temperatures fall rapidly below the initial freezing point. Numerous small ice crystals with filamentlike appearance are formed both intra- and extracellularly at approximately the same speed. Because of rapid rates of heat transfer, small ice crystals formed have little opportunity to grow in size.

Thus fast freezing causes spontaneous formation of many small ice crystals resulting in discontinuous freezing boundaries and very little translocation of water. Since most of the water inside muscle fibers freezes intracellularly, drip losses during thawing are considerably lower than in thawing of slowly frozen meat. In addition, muscle fiber shrinkage and distortion are minimized during fast freezing, resulting in near normal ultrastructure in the frozen state. Volume changes are less and periods of crystallization are shorter than in slowly frozen muscle, and consequently mechanical damage is correspondingly less. Filamentlike ice crystals entrap solutes and thus minimize the ion concentration effect. In addition, smaller and more numerous ice crystals in rapidly frozen meat reflect more light from meat surfaces, resulting in lighter color than that of slowly frozen meat (158).

Conditions under which frozen meat is stored are even more important for maintaining quality. The length of time frozen meat may be successfully stored varies with species, type of product, freezer temperature, temperature fluctuations, and quality of wrapping and/or packaging materials. In general, lowering storage temperatures may extend storage time of all types of frozen meat. Rates of chemical deterioration are greatly reduced by freezing, but reactions such as oxidative rancidity continue slowly even in the frozen state. Most chemical changes could essentially be eliminated by reducing temperatures to  $-80^{\circ}$ C, but such temperatures are not economically feasible in most storage facilities. Growth of putrefactive and spoilage microorganisms, and most enzymatic reactions, are greatly reduced, if not entirely curtailed, at temperatures below  $-10^{\circ}$ C. In general, storage temperatures of less than  $-18^{\circ}$ C are recommended for both commercial and home freezer units. Most of these units operate at temperatures between  $-18^{\circ}$ C and  $-30^{\circ}$ C. Although it is expensive to maintain the lower temperatures of this range, length of meat storage life may be significantly extended (158).

Temperature fluctuations during frozen storage should be avoided as much as possible, to minimize the ice crystal growth, the formation of large crystals, and the associated drip losses. Almost all water in meat is frozen at about  $-18^{\circ}$ C. However, as temperature increases, the unfrozen proportion increases and becomes especially marked above  $-10^{\circ}$ C. Small ice crystals are thermodynamically less stable than large crystals. Water molecules migrate from small crystals through unfrozen pools of water to recrystallize and form large ice crystals. Migration, recrystallization, and ice crystal growth are enhanced by relatively high storage temperatures and by temperature fluctuations. Fluctuating storage temperatures also may cause excessive frost accumulation inside packages, much of which is lost as drip upon thawing (158).

Acceptable quality may be maintained in frozen meat products for several months, only if certain critical packaging requirements are met. These requirements include the use of vaporproof packaging material to keep moisture in and oxygen out of packages. Moisture losses due to improper packaging materials or techniques result in dehydration and freezer burn, and oxygen from air in packages causes oxidative changes including rancidity. Another requirement for quality maintenance for extended periods of time is to eliminate as much air as possible from packages. Other requirements include the use of odorless greaseproof packing materials that are strong when wet and resist scuffing, tearing, and puncturing under normal handling conditions (158).

The amount of time meat may be held in frozen storage, while maintaining acceptable quality, depends on degree of saturation of meat fats. Since fish, poultry, and pork fats are more unsaturated than beef and lamb fats, they are more susceptible to oxidative changes. Hence recommended frozen storage times differ for various species. Gradual decreases in flavor and odor acceptability during frozen storage are primarily due

to the oxidation of lipids (158). The freezing process causes changes in the structure and color of the muscle. Storage temperature, light intensity, type of display area, and packaging all affect the rate of deterioration. The color of frozen meat is known to vary with the rate of freezing. Taylor (164, 165) found that, with lower freezing rates, the appearance of the product changed, and at very low rates, there was a marked development of translucence leading to a lighter product. Zaritky et al. (166) concluded that small crystals, formed by fast freezing, scattered more light than large crystals formed by slow freezing; hence fast frozen meat was opaque and pale, and slow frozen meat was translucent and dark.

The length of frozen storage is influenced by processing state of meat products, i.e., fresh, seasoned, cured, smoked, precooked, comminuted, or chemically preserved. Salt enhances development of rancidity, and processed meat products containing salt have limited frozen storage life. It is generally recommended that cured and smoked products not be stored frozen or stored only for very limited time. Sliced meat products such as bacon and luncheon meat should not be frozen unless vacuum packaged, because air incorporated during slicking, together with contained salt, lead to rancid flavor development in a matter of days or weeks. Precooked frozen meat and poultry products lose their "fresh cooked" flavor during frozen storage and develop a "warmed over" flavor due to lipid oxidation. These oxidative changes increase with cooking time but are minimized to some degree by treatment with polyphosphates, bases, or gravies. Oxidation is inhibited by pH increases caused by polyphosphates and by natural antioxidants present in many bases. Frozen storage life of precooked meat products also may be extended by packaging in inert gas, such as nitrogen, in which case elimination of oxygen from packages is responsible for the inhibition of oxidation (158).

Sensory quality after freezing in restructured meat products can be similar to that of processed meats. In the manufacture of restructured products, there are additional factors that may also accelerate the rate of discoloration, which is the main sensory quality altered by low-temperature storage. Of these factors, (a) grinding and mixing (167), (b) the addition of sodium chloride (168, 169), and (c) tempering of frozen meat logs (161) contribute also to a loss of sensory appeal. Serious problems, namely, color instability and fat oxidation, are encountered in restructured meats. Variable color patterns of restructured meat, especially beef products, continue to trouble processors, and color instability is a prime limitation to the acceptance of restructured meat products in the retail trade (170).

Cured meats develop rancidity more rapidly than frozen fresh meats. It is customary to freeze bellies or hams only before curing. In addition to rancidity, changes in flavor and texture occur in frozen cured meats. The relative stability of refrigerated cured meats decreases the need for frozen storage; however, the end user may desire to freeze portions of large products, such as ham. This can be done if the meat is properly wrapped and not held in the freezer for more than a few weeks. Longer storage periods lead to flavor changes (163). Mitchell et al. (171) found that while freezing and thawing steaks did not seriously affect the quality of the meat, steaks cooked directly from the frozen state were more juicy and had more flavor than those thawed first. However, they found that steaks thawed before cooking were slightly more tender than unthawed steaks, although there was little difference between steaks thawed in a microwave oven, in a refrigerator, or in air, a result also established by Moody et al. (172). The lower juiciness ratings for thawed steaks were attributed to drip loss during thawing (173).

The thawing process probably does greater damage to meat than freezing. Several factors are responsible for damaging effects occurring during thawing. Thawing occurs

more slowly than freezing, even when temperature differentials are the same. Further, temperature differentials in thawing are generally much less in practice than those used in freezing. During thawing, temperatures rise rapidly to the freezing point, then remain there throughout the entire course of thawing. This situation increases the duration of thawing compared to freezing and provides greater opportunity for the formation of new large ice crystals (recrystallization), for increased microbial growth, and for chemical changes. Thus time—temperature patterns of thawing are more detrimental to meat quality than those of freezing (158). The time required for thawing frozen meat depends on a number of factors: (a) temperature of meat, (b) thermal capacity of meat (lean products have higher thermal capacities and therefore thaw more slowly than fat products), (c) size of meat products, (d) nature of thawing medium (water provides faster heat transfer than air), (e) temperature of thawing medium, and (f) movement of thawing medium (158).

Factors affecting quality of frozen meat can be ranked in order of importance as follows: (a) frozen storage conditions, (b) thawing conditions, (c) freezing rate, and (d) prefreezing treatment and handling. Frozen storage, as conducted under commercial and household conditions, is the most damaging phase in handling and processing of frozen meat. Even though damage due to recrystallization of ice occurs during thawing, it is generally less severe than that occurring during storage because of shorter times involved. Original differences in product quality, caused by fast rather than slow freezing rates, gradually diminish during several months of frozen storage. In addition, cooking further reduces differences in quality caused by different freezing rates (158).

The single factor exerting the most damaging effects on meat products during frozen storage is temperature, particularly as it fluctuates. Deleterious effects produced by temperature primarily result from ice crystallization. Damage occurring during frozen storage can be minimized, but not entirely prevented, by maintaining low and constant storage temperatures, and by limiting the duration of frozen storage (158). During freezer storage an excessive loss of moisture from meat surfaces will result in localized areas of dehydration and discoloration. This condition is called freezer burn and is characterized by corklike texture and gray to tan coloration. It can result when wrapping and/or packaging materials have been punctured, or when moistureproof wrapping and/or packaging is not used. Improper maintenance of temperature, with frequent cycles of partial defrosting followed by refreezing during storage, also contributes to freezer burn. During the development of freezer burn, proteins become denatured and rehydration is poor. Meat with severe freezer burn is dry and tasteless or, if oxidative rancidity has developed, bitter in flavor (158). Freezer burn can be prevented by a skin-tight covering (moisture-impermeable film or dip or spray coatings) or an ice glaze (163).

Freezing may cause damage to meat structure, particularly if it is frozen too slowly. Water may collect extracellularly and freeze in large pools, forming spikes of ice crystals that puncture muscle fibers, releasing even more moisture. Physical damage caused by slow freezing results in great loss of fluid from meat when it is thawed. This fluid collects in packages upon thawing and is called drip. Excessive drip results in unattractive packages, loss of nutrients, and dryness in cooked meat (158). Profound effects on the structural and chemical properties of muscle foods, including changes in the muscle fibers, lipids, and proteins, result from slow freezing. All of these can significantly influence the quality attributes of meat and meat products.

Alterations in protein have been observed in frozen meat. Sarcoplasmic proteins are less soluble after freezing muscle tissue. The total extractable protein, sarcoplasmic protein, and actomyosin extractability decrease with frozen storage. Evidence indicates

that the quality of protein and fat deteriorate, and comminuted products manufactured from frozen meat block components gradually decrease in quality characteristics (163). While Miller et al. (174) found no acute emulsion instability during 37 weeks of storage, the trend indicated an eventual failure on further storage. The first signs of change were evident after only one week of freezing, and by 25 weeks all observed characteristics showed significant degradative changes.

During refrigerated storage, meat loses moisture from its surfaces, resulting in weight loss called shrinkage. Other than economic losses associated with shrinkage, loss of moisture during the first few days of refrigerated storage seldom has adverse effects on meat acceptability. However, physical changes accompanying shrinkage during prolonged refrigerated storage include surface dehydration and discoloration (158).

The texture and consistency of meat render it highly susceptible to the absorption of volatile materials. Meat tissues readily absorb aromatic compounds from other foods, such as apples or onions. Consequently, off-flavors may occur when meat is stored in the presence of such products (158). The beneficial effect of low-temperature storage is the tenderization of meat. Beef increases consistently in tenderness as freezing temperatures are lowered.

The morphology of ice in the frozen tissue and the size and distribution of ice crystals formed in the intra- or extracellular spaces are particularly important. These cause changes in the water-holding capacity of the muscles when thawed, in the texture, and in the surface color. Freezing of meat results in an increase in the amount of drip fluid when the meat is thawed, causing changes in the sensory properties of the meat after it is cooked.

The rate of freezing is of great importance, especially between  $-1^{\circ}\text{C}$  and  $-4^{\circ}\text{C}$  (30–25°F). When frozen slowly, ice crystal formation starts in the aqueous intracellular compartments. Subsequently, water in the connective tissue freezes owing to a lower salt content than muscle tissue. The remaining water is more concentrated, and the higher osmotic value will result in water diffusing out of the muscle cell and condensate on the outer cell ice. Eventually, the intracellular water will freeze. Under improper freezing conditions, ice crystals may grow to 150 µm (diameter) and damage the muscle cell (141). When the critical temperature ( $-1^{\circ}\text{C}$  to  $-4^{\circ}\text{C}$ ; 30 to 25°F) is passed rapidly (within 80–120 minutes), the water in the muscle cell freezes before substantial diffusion can occur. The existence of numerous small ice crystals limits the risk for cellular deformation. Fluctuation in storage temperature may permit the small ice crystals to thaw, leading to recrystallization and the formation of large crystals. This may lead to extensive membrane damage and subsequent problems with increased rancidity and drip or purge (141).

Deteriorative changes occurring during freezing are often owing to oxidation. The oxidation of meat during frozen storage is largely due to changes in the triglyceride fraction. Susceptibility to lipid oxidation is influenced by species differences, as well as differences between tissues. For example, beef lipids are more stable than those of chicken white meat. Temperature and length of holding can alter the rate of oxidation of lipids in meat. Even at  $-30^{\circ}$ C ( $-22^{\circ}$ F), lipid oxidation may occur. Oxidation during frozen storage is especially a problem with processed meat products, as salt acts as a catalyst in oxidative reactions. Auto-oxidation proceeds faster during storage at high temperatures. The susceptibility of frozen food systems to oxidation is related to low water activity. Whereas fresh meat has an  $a_{\rm w}$  of 0.99, muscle tissue frozen at  $-18^{\circ}$ C ( $0^{\circ}$ F) has an  $a_{\rm w}$  of 0.60. At water activities of 0.60–0.80, heme pigments appear to initiate lipid oxidation. Porcine myoglobin seems less stable to oxidation than myoglobin from other species. Other substances found in meat products are also known to accelerate oxidation.

Lipases and lipoxidases will, especially at a lower pH, induce lipid oxidation and rancidity (141).

Color deterioration of frozen meat can be a problem. Freezing accelerates metmyoglobin formation, causing an undesirable color change. Rapidly frozen meat is lighter in color than slowly frozen meat. Localized tissue dehydration, or freezer burn, may result in a lightening of dark tissue or a browning of light-colored tissues such as chicken skin. Freezer burn is an irreversible condition. It may be prevented by proper packaging. Even with proper freezing techniques, a concentration of soluble salts within the muscle cell will occur. This will cause denaturation of proteins, and the pH of the product will change (141). MacDougall (147) contended that a major color problem during frozen storage was photo-oxidation of pigments, indicating therefore that meat was best preserved by aluminum foil packaging. MacDougall's theory (147), that frozen meat oxidized from the surface inwards (compared to fresh meat, which oxidized from the interior outwards), can be demonstrated by the use of packaging techniques. Freezing of muscle foods should not start before rigor has set in. The primary textural change occurring in properly frozen red meats and poultry is an increase in porosity. Freezing and frozen storage of seafood can result in an undesirable textural change. The muscle may become tougher and drier and lose its water-binding capacity. The overall decrease in protein extractability is attributed primarily to alterations in the myofribrillar fraction (myosin-actinomyosin binding). The nature of the reactive groups responsible for the aggregation is not well understood. Formaldehyde, a product of the decomposition of trimethylamine oxide, is highly reactive with a variety of functional groups, and it has been suggested that it is involved in the covalent cross-linking of proteins in frozen-stored fish. The loss of water-binding capacity is probably due to an increase in the number of crosslinkages between myofibrillar proteins. As fish lipids contain highly unsaturated fatty acids, fish muscle is very susceptible to oxidation (141, 175).

Thawing of meats has been a largely ignored subject. Slow thawing will result in a better product than fast thawing, meaning that thawing at refrigeration temperatures is preferable to thawing at higher temperatures. Thawing time should not be too long, as it may lead to microbial growth. An important consideration in a thawed product is the amount of exudate. The exudate, containing soluble proteins, vitamins, and salts, will be increased when cell damage is extensive. Slow thawing allows for reabsorption of the fluids by the tissue (141).

Bulk frozen meat, typically in block form of up to 25 kg, can be processed immediately at a temperature of  $-20^{\circ}$ C to  $-30^{\circ}$ C or below. Heavy duty prebreakers in the form of guillotines, chisel-type implements, or strong cutter blades can rapidly reduce whole blocks into pieces suitable for further comminution (157). Similarly, heavy duty mincers or similar machines can operate at very low temperature and effectively reduce meat into small particles. However, the brittle state of meat and the ice contained within the tissues at these temperatures causes considerable shattering of muscle and fat structures, with the consequences of the meat being in a very finely fragmented state. This can be desirable for very fine comminutes of a homogenous character, but undesirable for products where larger particles and retention of muscle structure are required (157). The temperature at which the prebreaking of frozen meat blocks takes place can have a major influence on the ultimate particle size distribution of comminuted products, with meat prebroken at lower temperatures having smaller particles. The perception of particle size is itself a key determinant of burger texture (176). The breakage of meat and the considerable deformation of internal particle structure, often to the structural level of individual muscle fibers, is characteristic of mincing/grinding and

in turn determines the particulate character of many burger, sausage, and other meat products (157).

Beef could be satisfactorily preserved in the frozen state for at least 554 days at temperatures below  $0.9^{\circ}$ C. A later report (177) indicated that uncured pork cuts were in an excellent state of preservation after being stored for 9 months at  $-35.5^{\circ}$ C to  $-36.7^{\circ}$ C and that such cuts were still edible after 16 months under these conditions. A recent report (178) indicated that uncured pork chops and roasts could be stored at  $-30^{\circ}$ C for at least 196 days, regardless of the protective wrap used.

Frozen storage does not affect any of the surface or partial compression properties of meat. However, on the first bite fresh samples are slightly more cohesive and during mastication are chewier, require a greater number of chews, and are more cohesive than frozen and thawed samples. However, stored samples release slightly more moisture during mastication. After swallowing, the mouth coating from fresh samples consists of a higher proportion of particles, and the particles from fresh samples consists of a higher proportion of appropriate residual particles described as being fibrous, grainy, crumbly, and mealy, while the particles from stored samples consisted of a higher proportion of appropriate residual particles described as being fibrous and grainy. As a result of these differences, stored samples received higher texture amplitude ratings than their stored counterparts.

Present findings clearly indicated that freezing, frozen storage, and thawing produced beneficial effects on texture properties that enhanced the texture amplitude. Such effects resulted in a slight tenderizing effect, a slight improvement in juiciness, and a breakdown to more appropriate particles, as evidenced by stored samples being less cohesive, easier to chew, and releasing more moisture during mastication. However, it is also clear that freezing, frozen storage, and thawing produced detrimental effects that detracted from flavor amplitude. For instance, the higher moisture release during mastication observed substantiates previous findings that the juiciness of pork roasts improved slightly with freezing and frozen storage (178) and is in agreement with the results of Tuma (179) on beef. In addition, present findings that pork loins were less cohesive during the first bite and mastication, and were less chewy and required fewer chews during mastication, lends support to, and aids in explaining, previous reports that the tenderness of meat improved during freezing and frozen storage (180–184, 178). However, the low flavor amplitude ratings received by frozen and thawed samples clearly indicates that freezing, frozen storage, and thawing results in a less appropriate and less well balanced and blended flavor. Present findings also appear to be in agreement with previous conclusions that uncured pork palatability can be maintained for at least 6 months (177, 178).

Furthermore, present findings regarding moisture release, cohesiveness, and chewiness, coupled with the fact that frozen and thawed samples appeared to break down to more appropriate particles, clearly indicated that freezing and frozen storage is beneficial to the textural properties of pork loins. There is some evidence (185) that rapid freezing effects a greater tenderization because the ice crystals are formed within the muscle fibers, causing a disintegration of the fiber with an accompanying increase in tenderness. In one study comparing the eating quality of rib roasts of beef frozen under ten different conditions, it was concluded (186) that the more rapidly the meat was frozen, the better was its all-round quality. It has been claimed (187) that freezing is important in the maintenance of "bloom," or fresh appearance of poultry. This report refers to a "dark color which results from slow freezing." With a lower freezing temperature and hence a faster rate of cooling tenderness appears to be somewhat

improved. As far as the authors are aware, there is no satisfactory data to indicate that freezing, regardless of its rapidity, has an important effect on other palatability criteria in meats. Appearance, odor, and taste seem to be unaffected.

# H. Poultry and Eggs

## 1. Poultry

Flavor, texture and juiciness of smoked broilers stored at  $-18^{\circ}$ C for up to 12 months does not change (189). When broilers are fed with a diet containing rosemary and sage, and frozen stored at  $-20^{\circ}$ C, no rancidity is detected after 4 months (190). Chicken hot-drumettes containing 2% cayenne pepper are scored 2.5 after cooked (1 = no hotness, 5 = extremely hot) after four days of frozen storage (191). A duo–trio sensory evaluation of the flavor of breast meat from broilers fed with marine algae, vacuum packaged, and frozen stored at  $-23^{\circ}$ C showed no changes among fresh breast samples and frozen stored samples after 3 days of storage. Feeding 5.5% marine algae or 2.1% menhaden oil increase the flavor scores after 82 days of storage (192). Leg meat frozen produces more substantial oxidative decomposition of fats (193). The evidence indicates that juiciness, tenderness, and general palatability of broilers are the same whether the birds are frozen within 2 hours of slaughter or are held until the body temperature has dropped to the temperature of the cooler. Furthermore, as the period between slaughter and freezing is increased, the greater is the tendency for the fat in poultry to turn rancid during zero storage (194).

Frozen turkey breast does not show rancidity after 150 days of frozen storage. Oxidized aromas are normally detected. Color scores increase if the level of vitamin E in their diet is increased. In mechanically deboned chicken and turkey meat stored frozen for 6 months, some color is lost, but it is still acceptable for use in emulsion products (195). Chicken thigh meat patties formulated with 5 to 20% fat, cooked and stored for up to 9 months does not show rancidity (196). Patties made with ground dark turkey meat frozen and kept for 3 weeks are scored low if only polyphosphates or water are added, but those with alkaline tripolyphosphates receive higher sensory scores (197).

## 2. Eggs

Partial freezing, light freezing, and superchilling have been used to preserve the freshness of food products. Liquid whole egg, liquid yolk, and liquid yolk stored at -0.5 and  $-3^{\circ}$ C temperature for up to 50 days of storage do not affect the palatability test for scrambled eggs (198). Liquid eggs are used as ingredients in candy, baked goods, salad dressing, and ice cream. One-third of all pasteurized liquid egg is frozen prior to distribution to extend its shelf life. It has been reported that pasteurization and frozen storage influence liquid egg rheological and functional properties such as emulsifying properties, decrease in foaming, and whipping. Pasteurized frozen eggs stored for up to 80 days show changes in the solubility of certain proteins such as lipovitellin and livetin, which are responsible for the emulsifying properties of eggs (199, 200). The lumpy character of the thawed yolk is an undesirable quality that generally must be avoided. Egg yolk subjected to linear electron beam irradiation at  $\sim 2.5$  kGy dosage and frozen stored at -15°C for 60 days show that hue of irradiated samples is more yellow than the nonprocessed samples after 60 days of frozen storage. The saturation index is lower for the irradiated samples. Storage effects in redness and hue are also observed. The color of egg yolk becomes slightly less reddish yellow during storage or the yolk loses pigment during storage (201).

### I. Peanuts

Roasted peanut paste kept at -20 and  $-10^{\circ}$ C does not change its sweetness sensory attribute; however, bitterness (cause by saponins) and tongue burn tends to change over frozen storage. The most important sensory attribute that changes over frozen storage is "staling," used to describe light lipid oxidation and the characteristic flavor that results. This attribute has also been described as "old, cardboardy, strawberry-like note." As lipid oxidation progresses, changes are described as "painty." The fruity attribute in roasted peanut paste develops from abusive environmental and handling exposure but also increases over frozen storage. There is no change for the "roasted peanut" attribute during storage at low temperature (202).

# J. Soybeans and Soy Products

Food companies and consumers alike use the versatile raw ingredient known as soybeans in various food products for numerous reasons. Soy concentrates have the ability to bind and hold natural flavors and moisture, resulting in products that stay moist and flavorful even after reconstitution in conventional, convection, or microwave ovens (203). Soy flour has a blander flavor and is able to retain meat juices; while many lactose intolerant individuals consume soymilk. As a result, soybeans and their constituents are gaining popularity and usage in the food industry.

Soybean producers claim that consumers seek the sweetness, pleasantness, and firm, nutlike texture of the soybean. Young et al. (204) has reported that females are likely to purchase frozen soybeans to use in recipes as an alternative to meat. If improperly processed, however, soybeans and soy products develop objectionable off-flavors and offaromas. Bitter, astringent, and sour flavors are common flavor defects reported in soybean-based products. A chalky texture may be detected in some products, such as soy milk, owing to improper processing. In other soy protein products, the freeze-thaw cycle will result in a gummy and unacceptable product. These off-flavors and textures are important limiting factors in the use of soybeans and soy constituents in products that are shelf-stable and/or frozen. These off-flavors may be the result of the high proportion of unsaturated fatty acids found in raw soybeans. There are two major interrelated causes for the development of such flavors. One is the high proportion of unsaturated fatty acids, particularly linolenic acid, in the soybean oil fraction, and the other is the abundant presence of lipoxygenases in soybeans. The enzyme catalyzes the oxidation of polyunsaturated fatty acids of soy lipids, producing hyperoxides. Degradation of hyperoxides leads to the formation of various types of volatile compounds, many of which have an objectionable odor or flavor, described as beany or greeny.

Soy milk and soy products tend to have an objectionable aftertaste that has been described as sour, bitter, and astringent. These undesirable characteristics probably result from the presence of some minor compounds in soy milk including phenolic acids, saponins, oxidized phospholipids, and isoflavones. Soy milk, if it is not properly processed, tends to have a chalkiness defect. The freezing of this product tends to worsen this defect (205).

There are many different soy-derived products that are incorporated into foods for functional, nutritional, processing, and other properties. These ingredients can actually improve the nutrition, flavor, texture, and consumer acceptance of foods. If properly formulated, these types of soy-based or soy-enhanced foods freeze well with little detrimental attributes developed in the freezing process.

Soybean isolate is made from a spun soybean oil and is almost flavorless; its taste, therefore—exclusive of texture—depends to a large extent upon the quality and expertise involved in adding the flavors. Freezing has the tendency to make soybean isolates gummy. However, depending on the product in which it is incorporated, this may or may not make the product unacceptable. Soy protein isolates have been perfected that make possible a hot dog that contains only 3% fat, yet cannot be differentiated in flavor and texture from those one might be accustomed to and are competitive in price. These types of soy products tend to freeze well with virtually no loss of flavor or texture quality.

Extruded soybean extender, which can be in dry form as flour or pellets, often has off-flavors and must be blended with products or must have flavors or spices added that are strong enough to override the flavor of soy itself. Typically, this product freezes well, and because of the nature of the flavor system, it is not seen as being hindered by freezing. For example, there is a great deal of meatless chili made, both frozen and unfrozen, and the strong chili-powder flavor completely dominates the mixture. In this case, the soy extender is added to give the texture of the meat without the much higher cost of meat. There are even "beef" stews of this sort with no beef at all in them, but the generous quantity of spices present in all such stews drowns out the beany flavor of soy (206).

Formulating with textured soy proteins or such extenders is more complex than a direct substitution of another type of muscle food into a food system. Processors sometimes forget that meat is naturally about 80% water, and when an extender is used that absorbs too high a quantity of water it changes the texture and flavor of the beef by increasing the percentage of water to solids. If the product is frozen, this leads to an unacceptable product. Often, too, saturation occurs; the cells become ruptured, and the product becomes separated and soggy. This is an unacceptable quality attribute for products of this type.

Soy meat extender has also proven valuable in imparting proper texture to mechanically deboned meat. This product tends to freeze well, and no off-flavors or texture attributes are typically observed. Soy flour has the advantage of holding the juices in meats. Its disadvantage is that of taste and physical feel in the mouth.

Soy proteins are used extensively in breads, doughnuts, rolls, and sweet goods. They are not necessarily cheaper than the flour they replace, but they hold the moisture levels at an accepted and desired level, thereby producing a better-textured product. Some of those using it do not know whether to attribute the better texture to moisture retention or to some interaction of the soy proteins with the other ingredients that has not been adequately researched (203).

Soy-protein burgers, with the taste and mouth feel of beef, are an excellent source of protein. The grain-based burgers are usually higher in dietary fiber and are marketed to people who want a convenient food that has interesting textures and flavors, not necessarily imitating meat. The only caution is to avoid freeze—thaw cycles, because they make soy protein products gummy. However, most of these products are found in the grocer's freezer case, and are accepted well by consumers.

When used as emulsifiers, binders, moisture retainers, and stabilizers, soy proteins can alter or improve the appearance, taste, and texture of the finished product. Flavorwise, the white flake (soy flour and soy grits) has a bitter, beany taste. The toasted product is darker in color, and the enzyme and antitrypsin activities are destroyed (207). The flavor is also improved to the point where it takes on a sweet nutlike taste (208). The intermediate or cooked product has properties between the two.

Although the cost of soy protein concentrate is more than double that of soy flour  $(18\frac{1}{2}-28 \text{ cents per lb.})$ , it is rapidly replacing the soy flour used in emulsion products

because of its blander flavor. Soy protein concentrates tend to become oversaturated and are unable to retain moisture when incorporated into certain food systems and then subjected to freezing. Product shelf life can be shortened if the temperature of the freeze—thaw cycles is not carefully controlled.

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## 8

### **Texture in Frozen Foods**

William L. Kerr

University of Georgia, Athens, Georgia, U.S.A.

Freezing is used to preserve and maintain the quality of many foods. These include beef, chicken, pork, fish and other muscle foods; fruits and vegetables; egg products; dairy foods; doughs and breads; and a wide variety of entrée items. Relatively low temperatures, the formation of ice as a separate phase, and the freeze concentration of dissolved substances contribute to conditions that limit the growth of microorganisms and preserve quality factors such as flavor and color.

One critical quality factor influenced by freezing is food texture. Texture can be defined as those properties of food determined by the rheological and structural nature of the food and determined by the tactile senses. Many foods are thawed from the frozen state and eaten directly, or cooked before consumption. In some cases, the texture of the thawed material is close to that of the fresh and unfrozen food. In other cases, the texture may be changed by the freezing process and yet result in a thawed product that is still acceptable to consumers. For a few foods, freezing results in dramatic changes that are not acceptable to consumers. Yet another class of foods is those consumed frozen, such as ice cream, sherbert, or ice pops. In these, the food composition and particular freezing process have a profound effect on the perceived texture.

In this review, we will cover issues regarding the effects of freezing on the texture of major food groups. We will discuss primary texture attributes of particular foods and the fundamental mechanisms that reduce texture quality, and we will outline basic procedures before and during freezing that are used to ensure optimal quality of frozen foods.

#### I. VEGETABLES

Frozen vegetables make up a significant portion of the frozen food market. Vegetables that are frozen include potatoes, corn, peas, green beans, broccoli, cauliflower, brussels sprouts, and pumpkin. Freezing extends the availability of vegetables and preserves quality during transportation and storage. In some cases, it allows for more convenient processing of the food. For example, precut and treated French fries can be stored frozen for extended periods, with required portions available for immediate baking or frying.

#### A. Texture Attributes

The textural attributes of importance to most vegetables are firmness, tenderness, and crispness. Firmness implies that there is an immediate resistance to biting. Tenderness indicates that the material can be easily separated into smaller and smaller pieces, particular when sheared. Crispness is a complex attribute related to the sound and discontinuous breakdown of the product (1). In vegetables, firmness is directly related to turgidity in the vegetable cells (2). Turgor arises from cells that are fully hydrated and pushed back against the cell wall. Firmness also depends on the cell walls that surround each cell. A network of cell walls provides a semirigid scaffolding that holds the cells. The presence of lignin with thick cellulose layers, particularly in older plants, leads to a tough, woody texture (2).

Several physical tests are available for assessing texture in intact vegetables. Firmness is measured by the force developed as the sample is compressed. Measurements can be made through continuous readings of force and deformation or through a variety of penetrometers (3). Tenderness is often measured by shear force developed when a series of blades is run through the sample. Other specific tests such as the pea texurometer may be used.

#### B. Processing and Storage

The texture of frozen and thawed vegetables will never be better than that of the material before freezing. Vegetables with good flavor and texture should be used for freezing, and lots chosen so as to assure uniform maturity (4). After initial cleaning, many vegetables may be cut, sliced, or otherwise processed prior to freezing. Such commodities include sliced or diced carrots, French fry cut potatoes, and corn without the cob.

Most vegetables require blanching prior to freezing. Blanching is the use of hot water or steam to deactivate enzymes in the vegetable (5). Several enzymes can cause undesirable changes in the frozen vegetable. For example, lipases and lipoxygenases lead to the formation of off-flavors (6). With respect to texture, pectin-hydrolyzing enzymes cause excessive softening of the tissue during frozen storage. Blanching has the added benefit of reducing microbial load and internal gases.

Unfortunately, significant changes in texture are induced by heating during blanching (7,8). Blanching disrupts the normal cell structure. The cell membrane may be damaged and allow water to enter the cell. Internal organelles may be distorted and begin to leak their contents. Heating may also denature proteins as well as gelatinize starch found in cellular granules. This starch may subsequently retrograde during frozen storage. The major impact of blanching on texture is the reduction of cell turgidity caused by the loss of cell membrane function. When cells do not push up against the cell wall, the tissue becomes flaccid and less firm. In addition, during blanching, pectic substances are released from the cell wall. This causes less cohesion between adjacent cells and thus a breakdown of the structure.

Blanching methods and times depend on the particular vegetable, and in some cases on the particular cultivar. In general, it is advantageous to use cultivars that require minimal blanching, thus reducing textural changes caused by heating.

There is some evidence that the particular blanching regime affects the texture of the thawed product (9, 10). Brussels sprouts preheated to 52°C subsequently required 20% less blanching (9). Carrots blanched at 76°C were firmer than those blanched at 100°C (11).

Similarly, green beans blanched at lower temperatures were firmer and exhibited less sloughing (11).

There have been some studies on changes occurring after blanching. The breakdown of pectins is enhanced in high acid conditions (12, 13) and continues at neutral pH. Firmness may be enhanced by the interaction of demethylated pectins and divalent cations. Added calcium may help increase firmness in some vegetables (13, 14). Low-temperature blanching may increase calcium availability or pectin methylesterase activity (15–17). Subsequent linking of demethylated pectin by calcium increases firmness.

For most vegetables, rapid freezing results in optimal texture. Rapid freezing allows less time for osmotic dehydration of cells and the associated freeze-concentration of solutes. The particular freezing method depends on the type of vegetable, its size, and its structural features. Air blast and cryogenic freezing methods have been used successfully with most vegetables. In limited cases, freezing in blocks may be warranted.

#### II. FRUITS

Many fruits are frozen to extend their availability throughout the year, and to further shelf life through lengthy distribution. In general, fruits have a less fibrous structure than vegetables and often suffer more textural changes during freezing and storage (18). Thus direct consumption of frozen/thawed fruits is not as substantial as that of vegetables. Many frozen fruits are destined for further processing, as for use in muffins, ice cream, yogurt, or jams. In addition, large whole fruits do not freeze well. Fruits such as apples, peaches, melons, or pineapples are typically cut into slices, chunks, or segments prior to freezing. Smaller fruits such as blueberries, strawberries, blackberries, or grapes may be frozen whole.

#### A. Textural Attributes

As with vegetables, firm yet tender fruit are desirable. In general, freezing does not improve fruit texture, so that fruits that are hard, mealy, too soft, or weepy should not be frozen, particularly if they will be consumed directly. Loss of water holding capacity is also a problem for many frozen fruits. Such fruits exhibit excessive drip loss on thawing, and may lack proper juiciness when chewed.

#### B. Processing and Storage

Of course, the quality of raw ingredients is important to the texture of frozen/thawed fruit. In some cases, the freezing of slightly unripe fruit results in a firmer texture with lower drip loss (19, 20). For example, underripe apricots, cherries, and plums had lower drip loss after freezing and thawing (20). Similar results were seen in slightly unripe blackberries (21).

The fruit variety may also be a factor in freezing performance. Differences in the texture of thawed fruit due to variety differences have been found for strawberries (22, 23), red and black currants (24, 25), and raspberries (26). However, the maturity and grade of the fruit are probably more important factors than cultivar differences on the texture of thawed fruit (27, 28).

Immediate freezing of fruit after harvest can also improve texture. For example, strawberries frozen within 2 hours of picking, and stored for 3 months, had better texture than those frozen after 5 hours (29). In some cases, harvested fruit may be stored for

longer times at lower temperature and in modified atmospheres prior to freezing without affecting textural quality (19, 29).

Fruits are seldom blanched before freezing. Any benefits to be had by blanching are outweighed by the significant cellular damage it causes. Fruits do not have the more extensive fibrous network of vegetables, and therefore become mushy or even disintegrate with heating. There are some exceptions to the rule, however. For example, polyphenol oxidase can be inactivated in bananas by blanching, without causing unacceptable changes in firmness (30). Similarly, blanched breadfruit segments had acceptable texture when thawed and cooked (31). Some work has also been done on nonthermal blanching of fruits. For example, gas mixtures with sulfur dioxide have been shown to inhibit enzyme activity in sliced and frozen apples (32).

Many fruits are cut or diced prior to freezing, and have pits or stones removed. As freezing rate is of particular importance to the quality of frozen fruit, pieces with the shortest dimension greater than 2–3 cm are not typically frozen (33–35). Freezing times increase somewhere between linearly with or as the square of the shortest dimension for heat transfer. In addition, much fruit is used for further processing, and fruit cut prior to freezing is often easier to cut and of better quality than fruit cut after freezing and thawing (36, 37). Removal of pits in apricots, plums, and cherries may cause greater drip loss in thawed fruit, but may be necessary for other reasons (20, 29).

Various ingredients may be added to enhance the quality of frozen fruits. Sugar is often used to improve color, texture, and flavor (38, 39). Water is drawn out of the cells by osmosis, which leads to a lower freezing point and a decreased fraction of ice at a given frozen storage temperature. An extension of this is osmotic dehydration in which high concentrations of sugar solutions are used. With this approach, fruit cells may lose up to 40–70% of their water. Fruits such as strawberries, apricots, cherries, and pineapple have been subjected to osmotic drying prior to freezing (40, 41). In general, such fruits are sweet and firm and have lower drip loss.

The formation of extracellular ice concentrates the solutes in the surrounding aqueous phase, which in turn drives water from the cells. The higher concentration of intercellular solutes, coupled with the lessened flexibility of the frozen phase, produces cell and cell membrane damage. In addition, cell wall substances, particular pectins, may be extracted or solubilized from the cell walls, reducing structural rigidity.

Various freezing methods, including air-blast freezing, fluidized-bed freezing, and cryogenic freezing have been used successfully with fruits. Individual quick frozen (IQF) methods are preferred. Contact plate freezing of fruit in blocks is used little in current practice. A significant body of work exists to show that rapid freezing results in the best retention of quality in thawed foods (36, 37, 42, 43). This is especially important in frozen fruits, which are subject to greater textural changes in the freezing process. Rapid freezing results in smaller ice crystal size, less cell dehydration, and less cell damage (37, 42). Comparative studies on mangoes (44), strawberries (45), apples and peaches (46), and blueberries (47) have shown that rapid freezing rates result in thawed products with the best texture and least drip loss. Cryogenic freezing with liquid nitrogen often results in the best texture. However, prolonged immersion in cryogens may cause "freeze fracturing" at the surface (48, 49). Sprays of liquid nitrogen generally cause less freeze-fracture damage than immersion (50).

#### III. MEAT

Many muscle foods are frozen to preserve quality, including red meats (primarily beef and pork), fish and other seafoods, and poultry. Properly frozen and stored meat will maintain good texture and flavor for many months. All muscle foods are high-protein foods with cells containing an orderly sarcomere structure, and with tissues held together by membranes and collagenous material. Each requires the proper mix of firmness and tenderness for optimum texture, as well as the retention of moisture for optimum juiciness. Unlike fruits and vegetables, virtually all meats are cooked before consumption, with the one goal of affecting muscle texture. This occurs as sarcomeric proteins are denatured and solubilized and form soft gel networks, and as collageneous materials melt and reduce muscle toughness (51). Differences in muscle type, metabolic processes, and production practices exist, and they dictate variations in freezing practices. In this section we will discuss texture changes during freezing of red meat, with fish, seafood, and poultry meats covered in subsequent sections.

#### A. Texture Attributes

In general, meats have a more challenging texture than vegetables or fruits. Intact meats should be relatively firm and cohesive and offer a certain amount of bite resistance. However, cooked meat should be tender enough that excessive chewing is not required. Meat toughness is largely dependent upon the properties of the connective tissue proteins (52). For example, as an animal ages, irreversible cross-links develop in the collagen and cause the meat to be tough.

Muscle tenderness is usually measured by the amount of force required to shear through the meat. In the Warner–Bratzler shear method, cylinders of cooked meat are placed in a holder. A knife blade with a triangular opening is passed through the sample, and the maximum force attained before the sample is cut through is used as the measure of tenderness (3). Alternate methods exist such as the electric penetrometer and correlations with trace-metal analysis (53).

Quality meat should also be juicy and have maximum water-holding capacity (WHC). Meats lacking WHC are dry and tough and have unsightly losses of liquids during thawing and cooking. Thaw and cook loss can be quantified by weighing before and after thawing or cooking and calculating the drip loss by difference. WHC can also be measured by compressing cooked samples, such as 1 cm cubes, by a given force or deformation, and weighing the amount of liquid expressed onto filter paper.

#### B. Processing and Storage

The biochemical state of meat is one of the prime factors that determine quality after freezing. After slaughter, most meat must undergo aging and conditioning before being frozen. Much of the meat quality is related to pH in the muscle. As muscle ages, glycogen in the muscle is converted to lactic acid, which in turn reduces muscle pH. The meat is aged in a cold room so that an ultimate pH will be attained (54–56). An ultimate pH of about 5.6 is attained within a few days and results in the best meat texture. Typically the process is speeded up by electrical stimulation of the carcass (57).

Animals with low glycogen levels may result in meat with higher ultimate pH, which in turn causes a dark, firm, and dry (DFD) meat (58–60). In addition, if muscle pH remains high during cold storage, "cold shortening" of the muscle may occur (61). Such meat is

very tough and often unacceptable (61, 62). Shortening is particularly a problem in meat that has been removed from the bone (63) but can be minimized with proper cooling (64). Conversely, if muscle glycolysis occurs too quickly, a pale, soft, exudative (PSE) meat will result (65), particularly in pork. PSE meat has high drip loss before and after freezing, resulting in colorless dry meat that lacks juiciness.

As found with most foods, rapid freezing results in an optimal texture of the thawed, cooked meat. In some cases, freezing may even increase the tenderness and juiciness of the final product (66). Slow freezing results in larger ice crystals, greater cell dehydration, more extensive cell damage, and greater protein denaturation (67–69). The movement of cell water to extracellular spaces during slow freezing results in decreased water-holding capacity. Such meat is more likely to incur thaw and cook loss, and consequently to be less tender and juicy. Freezing times less than 20 minutes were found to produce the least exudates in frozen/thawed meat (70). The amount of time that the product spends at temperatures near  $-3^{\circ}$ C may be particularly important, as the freeze-concentration of ions at this temperature produces greatest protein denaturation (69).

Air-blast, cryogenic, and contact plate freezing have all been successfully used for meat freezing. Fast freezing rates attained with cryogenic freezing may be advantageous, but such methods are limited to meat pieces of modest volume and that can be individually exposed to the cryogenic fluid.

Frozen meat may also incur a drying of the meat surface known as "freezer burn." Although a slight amount of surface drying is useful for limiting microbial growth, excessive drying results in a pale, dry-looking surface with greater toughness and reduced juiciness. Freezer burn may occur during freezing and is a particular problem during extended frozen storage. It is enhanced by low humidity and fast moving air, and meat with less surface fat tends to suffer more surface dehydration (71). Appropriate wrapping can help limit the occurrence of freezer burn (72, 73).

Other textural changes can occur during frozen storage, and storage temperatures below  $-18^{\circ}$ C are recommended to limit these. Ice recrystallization is a problem that encourages protein denaturation, subsequent toughening, and loss of water-holding capacity. Higher temperatures, and in particular repeated temperature fluctuations, are detrimental to texture quality.

Thawing methods can also affect the final texture of the meat. Unfortunately, not many thawing alternatives exist. Rapid thawing, particularly through temperatures near  $-5^{\circ}$ C to  $0^{\circ}$ C, prevents further toughening and loss of WHC (70). In some cases, thawing and holding the meat may help improve tenderness. By this logic, thawing in a cooler or refrigerator would seem not to be recommended. However, thawing at higher temperatures may result in parts of the product reaching elevated temperatures before all of it has thawed. This would subject the meat to greater microbial spoilage. Thawing by direct immersion in water can be beneficial. In a few instances, meat may be cooked directly from the frozen state, but the possibility for uneven cooking or uncooked centers limits this approach. Research on vacuum, dielectric, infrared, and microwave thawing are ongoing.

#### IV. FISH AND SEAFOOD

Although fish and seafood are muscle foods, they have unique characteristics that distinguish the way they are processed for freezing. First, harvesting of particular fish has more seasonal variability, so that freezing becomes necessary to ensure availability

throughout the year. In addition, fish are taken at locations remote from where they are frozen and stored. Fish also have shorter muscle fibers and less connective tissue than land animals and are expected to be more tender than red meat and poultry. Different metabolic processes also lead to more substantial flavor changes in frozen fish.

#### A. Texture Attributes

Changes in flavor are perhaps the greatest concern in frozen fish. Fish have different muscle fibers and do not have the large skeletal system and connective tissue associated with land animals. Fish meat is generally expected to be firm but tender, and remain juicy and succulent. Some changes in texture do occur in the freezing process and are often associated with protein denaturation and drying out of the meat.

#### B. Processing and Storage

Fresh fish are often chilled, placed in ice, or frozen while on board ship. Ice glazes using salts, phosphates, antioxidants, or sodium alginate help prevent surface drying and lipid oxidation during transport (74). Moisture-proof packaging is important to prevent dehydration, and vacuum-packaging is useful for limiting lipid oxidation (75). In some onboard operations, fish are individually placed in plastic bags and vacuum-sealed, and then frozen in brine freezers (76).

Like red meats, fish and seafood should be frozen as rapidly as possible. Slow freezing results in cell dessication and damage that results in toughness and lack of moisture retention. Fish is especially subject to protein denaturation during freezing and frozen storage (77–79). Freeze concentration of solutes enhances the denaturation of myofribillar proteins. Some fish, in particular cod and deepwater fish, contain trimethylamine oxide (TMAO). TMAO breaks down to dimethylamine and formaldehyde in frozen storage, creating protein cross-links that produce a spongy texture (74). Such fish release moisture quickly, leaving behind a dry, tough texture.

#### V. POULTRY

Poultry is another excellent source of protein, and the poultry market has enjoyed substantial growth in the last few decades. Much of the chicken and turkey sold these days has been further processed. Most chicken is sold in cut pieces rather than as whole birds, and it often incorporates phosphate or other marinades. Turkey is sold as whole birds or in pieces and is usually injected with salt and phosphate solution. According to USDA regulations, poultry that is held at temperatures below 0°F must be labeled as "frozen," while "fresh" poultry is that held at temperatures above 26°F.

#### A. Texture Attributes

As with other muscle foods, poultry meat is expected to be tender and juicy. Loss of juices during thawing can be a particular problem. Meat from different parts of the bird typically has different textural characteristics. Red meat has more fat, is generally less dry, and has more lubrication when chewed. White meat has more of a tendency to be dry and tough if improperly processed. Simple tests are used to assess chicken texture, like those used for

red meats, including Warner-Bratzler shear tests, water-holding capacity tests, and analysis of yield, drip loss, and cook loss characteristics.

#### B. Processing and Storage

As with all products, the quality of the commodity before freezing is perhaps the most important factor determining final texture. The USDA provides grades (A, B, C) which may or may not be indicators of good texture in the cooked meat (150). The age of the bird at slaughter is a determinant of texture (80). Young "broilers" tend to have more tender meat than older hens. Similarly, young turkey fryer-roasters have more tender meat than older hens and toms.

Rough handling during processing may result in carcass damage, but the effects on texture are not known. In general, poultry must be chilled to  $40^{\circ}$ C within 4–8 hours to meet USDA specifications. Although used primarily to limit microbial growth, rapid chilling also ensures optimum tenderness. During ice-water chilling, moisture may be absorbed, but it must be limited to less than 8% for chicken and 4.3% for turkeys. This water in turn may increase freezing times and influence final juiciness. In general, freezing immediately following slaughter is not recommended. As with other meats, poultry should be aged to allow development of the ultimate pH, which results in the most tender meat (80, 81). For chicken, the hold period is 6–8 hours while for turkey it is 12–24 hours (82). The aging time for poultry is much shorter than that for red meat, and some studies suggest that the prefreeze hold time is not a major factor for quality after frozen storage (83). Generally, poultry should be frozen to less than  $-18^{\circ}$ C within 72 hours (81). Although not strictly freezing, there has been a move towards deep-chilling of poultry to -2 to  $-3^{\circ}$ C, particularly for younger broilers.

A wide variety of further processing operations are performed on poultry including deboning, chopping, forming, and breading. Poultry must be raw or fully cooked prior to freezing. Operations that slice, cut, or expose more surface area allow for greater drip loss. However, drip loss may be controlled in such products. In particular, marination combined with tumbling or injection has a profound affect on the yield, tenderness, and juiciness of poultry meat (51). The solubilization of muscle proteins and breakdown of sarcomeric structure result in lower shear values, enhanced swelling of myofibrils, and greater water holding capacity.

Appropriate packaging is critical to maintaining quality during freezing (84). In addition to preventing oxidation and flavor changes, moisture barriers help prevent dry meat by limiting surface dehydration and "freezer burn." In general, vacuum packaging or gas flushing is used to limit flavor changes, while also preventing overly slow freezing.

As with other muscle foods, rapid freezing provides the best product quality. Excess time spent at subfreezing temperatures is detrimental and leads to protein denaturation that results in toughening and drip loss. Rapid freezing also helps ensure a light, chalky surface (85).

Poultry is frozen by a variety of methods including air-blast, cryogenic, and immersion freezing. As long as the freezing rate is sufficiently rapid, there does not seem to be much affect on meat texture for different freezing processes (81, 86). Some research has shown that tenderness in chicken breast is not dependent on freezing rate (87, 88), but rapid freezing may decrease the amount of drip loss (89–91). Fried broiler pieces frozen with liquid nitrogen showed greater tenderness than those frozen in air (92). It has been recommended that the freezing front progress at rates greater than 2–5 cm per hour (82).

Combination freezing, that is, initial freezing with liquid nitrogen or by immersion followed by air-blast freezing at less than  $-30^{\circ}$ C, may be advantageous.

If properly stored, some poultry meat may be kept in frozen storage for up to 2 years without significant deterioration of quality (93). Storage temperatures should be below  $-18^{\circ}$ C for optimum quality. Some studies suggest that drip loss may increase during extended frozen storage (94). As with other frozen foods, large temperature fluctuations are particularly detrimental. The resulting recrystallization and formation of large crystals leads to toughening and drying of the meat.

Preprocessing and freezing seem to be greater factors than thawing method on drip loss and tenderness. Thawing of whole turkeys is especially of interest. However, studies have shown that different thawing procedures have little effect on texture and moisture retention (95, 96).

#### VI. EGGS

Egg products are used extensively in restaurant and fast-food establishments, and as an ingredient in baked goods and entrees. Much of the eggs are prepared at an egg-breaking facility, then dried, cooked, or pasteurized and frozen. The resulting products are frozen liquid eggs or frozen cooked eggs. Eggs lack the fibrous structure of vegetables and muscle foods, so are not subject to the same deterioration of structure. Although the physical process of freezing is the same, eggs have unique characteristics that determine how they are processed and frozen. The freezing of eggs is governed by USDA regulations.

#### A. Texture Attributes

In products in which eggs are a minor ingredient, the texture of eggs per se is not a major issue. However, the functionality of egg proteins in such products is important, as it may determine foam or gel structure and emulsification properties. As liquid products, thawed, uncooked eggs are characterized by their viscosity, and the viscosity is usually a good indicator of the suitability of liquid eggs for further processing. The texture of eggs cooked from frozen liquid eggs should be similar to that of unfrozen eggs. In general, cooked eggs should be firm but tender and have adequate cohesion and little run-off. The association of egg white proteins produces a gel network. However, excessive cross-linking may produce an undesirable rubbery texture.

Eggs have clearly distinct phases, that is, the egg yolk and egg albumen, and these have unique chemical compositions. The albumen is approximately 89% moisture, 10% protein, 0.5% carbohydrate, 0.5% ash, and 0.03% lipid (97). The yolk contains 48% moisture, 16% protein, 0.8% carbohydrate, 1.1% ash, and 33% lipid. In addition, the functionality of protein and lipid differs in each region. Compositional factors produce differing rheological properties for egg yolk and albumen, while functional properties also dictate the textural characteristics of products made from eggs or egg components.

#### B. Processing and Storage

Most preprocessing of eggs includes washing, breaking of the shell, and relatively low-temperature pasteurization. In addition, yolks may be separated from the whites and then later remixed or sold as separate products. Owing to compositional differences in egg yolk and albumen, the effects of freezing are different for each.

Liquid egg white has a relatively high level of solutes, including protein, glucose, and salts. Freeze concentration during freezing produces high solute concentrations and ionic strength. This in turn may cause denaturation of the egg white proteins (98). The concurrent aggregation of denatured protein reduces functionality. However, egg whites are not greatly changed by the freezing process. Cakes and other baked goods made from frozen/thawed egg white have volumes similar to that made from unfrozen egg white (99). However, gels made directly from frozen/thawed egg white are firmer than those from unfrozen egg white (100). Viscosity and denaturation depends somewhat on the freezing rate. Rapidly frozen egg white has a viscosity, foam stability, and native protein content closest to that of unfrozen eggs (101). Lower freezing rates result in greater denaturation, lower viscosity, and less foam stability. In some cases, adjuncts may be added to modify the rheological properties of egg albumen. The addition of 5–10% sucrose helps to prevent the loss of protein functionality caused by freezing. Gels made from egg white with added sugar have higher gel strength and elasticity (102). The addition of 5–10% NaCl, however, reduces gel strength, elasticity, and viscosity.

Egg yolk undergoes greater changes in texture during freezing than does egg albumen. The yolk contains granules and lipoprotein micelles that can become unstable and aggregate. Freezing causes thickening and partial gelation of the yolk (103, 104) at freezing temperatures below  $-6^{\circ}$ C, with maximum gelation occurring at temperatures below  $-18^{\circ}$ C. The degree of gelation increases during frozen storage. Textural changes occur as freeze concentration of salts and changing pH produce protein denaturation (105–107). Gelation has also been attributed to interactions between granules and low density lipoprotein (108).

While deterioration of egg yolk is ameliorated by rapid freezing, storage at low temperatures increases the amount of yolk gelation (108). Gelation is also reduced by the addition of low-molecular-weight solutes such as sucrose, NaCl, or glycerin (109, 110).

Liquid egg products are generally frozen in cans or pouches in air-blast freezers. Cryogenic freezing with liquid nitrogen or carbon dioxide provides more rapid freezing, but it is used mostly for cooked egg products. These are generally flat, thin sandwich eggs or omelets. There has been some promising research on drum freezing of liquid eggs to form flakes (98).

#### VII. DAIRY PRODUCTS

Unlike fruit, vegetables, and muscle food products, most frozen dairy products are eaten while still frozen. Such products include ice cream, sherbert, and frozen yogurt. Products such as milk, cheese, or butter are typically refrigerated and consumed within relatively short periods. However, in some cases dairy products are frozen in order to extend shelf life.

#### A. Texture Attributes

Desirable texture attributes vary with the type of dairy product. The chemical compositions of milk, cheese, yogurt, butter, and frozen desserts are narrowly defined. Processing methods are also usually standard, so that uniform texture is expected for most dairy products. Liquid milk is typically expected to have a certain viscosity and mouth feel, be free of aggregated material, and have some cling in the mouth. As protein and carbohydrate content are similar in most milk, the serum viscosity does not vary much.

Fat content plays a more important role to viscosity. Milk with more fat has greater viscosity and better mouth feel. The degree of homogenization has some effect on viscosity. However, most homogenization schemes produce particle sizes between 0.25 and  $3\,\mu m$ . Milk with much larger particle sizes would tend to separate. Milk with higher average fat particle size will have higher viscosity. Changes in particle size that occur through aggregation or coalescence will have a profound effect on the fluid rheology.

Cheeses have perhaps the most textural variety. The firmness, cohesiveness, adhesiveness, and chewiness vary with the moisture and fat content, the type of milk used, and the preparation procedure. For example, cream cheese is expected to be soft, smooth, cohesive, and spreadable. Hard cheddar cheese should be firm, less cohesive, and more springy.

Ice cream may also have some textural variety. An ideal ice cream should be cold but not "icy," firm but not too hard, have a smooth body and meltdown, and be free of any grittiness. Some textural differences are expected, for example, in "soft-serve" ice creams or frozen shakes. Some differences in texture may be attributed to price and composition. Premium ice cream has higher fat (14–18%) content and lower levels of stabilizers. Shorter shelf life and more stringent distribution channels may be needed to ensure product stability. Other ice creams have at least 10% fat and may incorporate a variety of stabilizers, sugars, and dextrins. These help prevent changes in ice crystal structure during frozen storage, but may contribute to stickiness or unexpected meltdown characteristics.

#### B. Processing and Storage

Very little fluid milk is frozen for storage. Concentration of milk prior to freezing increases the efficiency and improves the quality of frozen milk (111). Significant textural changes can occur in frozen milk (111, 112). Due to freeze concentration, casein micelles tend to precipitate or coagulate, resulting in increased serum viscosity. In addition, fat separation can be significant, and fat must be redispersed after thawing. The texture of milk has been found to deteriorate in frozen storage (113). When stored at  $-10^{\circ}$ C, the texture had deteriorated significantly after 2 months of frozen storage.

There is a greater potential for freezing of high fat cream for use in other products. Cream with more than 25% fat can be frozen with minimal separation. The addition of up to 10% sucrose increases stability. The use of drum freezing to produce flaked cream has been researched (114). The texture of butter is little affected by freezing, although lipid oxidation may occur at storage temperatures above  $-20^{\circ}$ C (111). Cheese can also be frozen to good effect, particularly cheese to be used on pizzas or entrees that are cooked or baked. Frozen and thawed cheese has a tendency to be more "crumbly" than unfrozen cheese (115).

Ice cream and dairy desserts comprise the major portion of frozen dairy products. Ice cream is a complex food in which air is incorporated in a composite of ice, unfrozen aqueous solution, and oil-in-water emulsion. The formulation and processing of ice cream has a profound effect on the final texture. Higher fat content produces a rich, smooth texture and body, with excellent melting properties (116). During freezing, the protein-stabilized fat membrane can disrupt, producing coalescence with other fat particles. A modest amount of coalescence can increase smoothness and richness. Excessive coalescence is produced when there is too much shear. The resulting ice cream has a "churned" quality reminiscent of butter.

The nonfat solids include milk proteins, lactose, minerals, and other components. The surface active milk proteins help stabilize the relatively small  $(0.5-2\,\mu\text{m})$  fat globules produced during homogenization (117). The proteins also contribute to serum viscosity and help stabilize the foam structure produced during initial freezing (118).

To produce ice cream, homogenized and pasteurized ice cream mix is placed in a swept-surface freezer to reduce temperature and remove the heat of fusion (119). Air is incorporated into the product by whipping or by direct injection of compressed air. The amount of air, or overrun, in the product, as well as the air bubble size, have an important effect on ice cream texture. Up to half the volume of the frozen ice cream may be air. Increased air provides a lighter, less dense texture. Generally smaller, more uniform air bubbles produce better texture (119).

After approximately 50% of the water in the product is frozen, the soft-frozen ice cream is placed in packages and placed in hardening freezers. Once the ice cream reaches the consumer, perhaps the most important determinant of texture is temperature. At progressively lower temperatures, more and more water exists as ice, and the ice cream becomes progressively harder. As the temperature is increased towards the freezing point, the ice cream becomes softer.

Several defects in texture may occur during frozen storage. Crystallization of lactose causes a gritty texture known as "sandiness." Lactose often exists as a supersaturated solute in the unfrozen phase. Crystallization is limited by control of diffusion by high viscosity (120). In this sense, the incorporation of whey protein and hydrocolloid stabilizers may help limit sandiness. Current stabilizers include carageenan, locust bean gum, gelatin, guar gum, and sodium alginate (121–123).

Iciness is another defect in texture that is manifest during frozen storage. Small ice crystals produce smooth body in ice cream but unfortunately are inherently unstable. Larger crystals produce a texture that lacks smoothness and is overly cold. At constant temperature, Ostwald ripening causes migration of water from small crystals to form larger ones. Temperature fluctuations are even more detrimental to texture. As the temperature rises, ice melts. As the temperature is lowered, this water refreezes slowly to form relatively large ice crystals. Stabilizers provide some protection against recrystallization and subsequent iciness (124, 125). Presumably, stabilizers limit diffusion of water, preventing continued growth of large crystals in neighboring regions.

#### VIII. BAKED GOODS

A variety of doughs and baked products are made from cereal-based flours. In the U.S., bread is a major staple of the diet. However, the flavor and texture of bread deteriorates quickly, and it would be advantageous to develop processing systems to extend shelf life. Freezing is one technique that has had some success in preserving the quality of dough and bread. Currently, it is more advantageous to freeze dough than to freeze bread. With the advent of in-store baking in supermarkets and fast food restaurants, the accessibility of frozen dough allows for continuous production of fresh bread.

#### A. Textural Attributes

Bread has a semisolid foam structure that incorporates different levels of air depending on the bread type. Dense, heavy breads have less air cell structure, while light, fluffy breads have more air cells. Bread is expected to be relatively soft and easy to shear but firm enough to resist the forces of spreading. Bread is particularly susceptible to changes in texture due to staling. Linked primarily to the recrystallization of starch, staling causes a firm, tough, and dry texture. Firmness is normally measured by the maximum resistance developed when a cylindrical probe compresses a bread sample.

#### B. Processing and Storage

When freezing dough, the most important consideration is retention of yeast activity. After thawing, the dough should expand as carbon dioxide is formed from yeast fermentation. This provides the finished bread with a lighter, softer, less dense texture. Curiously, slow freezing is the least harmful to yeast activity (126–128). Although slow freezing produces more yeast cell dehydration, it has been proposed that this helps protect the cells from freeze damage (127). It has also been found that freezing should be done as soon as possible after dough formation (129, 130). Dough systems in which fermentation has commenced prior to freezing have lower yeast activity after thawing. Yeast strains with greater freeze—thaw tolerance have been developed for use with frozen dough (131). Rapid-rise yeast does not withstand freezing as well as slower rising varieties. Adding higher levels of yeast initially may help make up for loss of activity during freezing and frozen storage (132). Loss of yeast activity increases with time in frozen storage.

Weakening of the gluten network is also a problem during freezing and frozen storage of dough. This adds to the problem that less time is allotted for proofing of the dough. Weakened dough is less able to retain the gases produced during fermentation. One way to mitigate the effects of dough weakening is to add more protein to the initial mix. French bread made from 12.8% protein, as opposed to 11.1% protein, had better dough properties and texture in the finished bread (133). Protein content may be increased by using a "harder" wheat, or by adding vital wheat gluten. A weaker dough has also been associated with high amylase flour (132) as well as excessive dead yeast cells (135, 129). Using 3–5% less water in the dough mix can also help improve dough quality.

Nonflour ingredients also affect the dough properties. Oxidants can help stengthen the dough (132, 136). Adding higher levels of shortening can help improve the quality of frozen dough (137). Surface-active agents can also improve dough properties, and subsequently loaf volume of the baked bread (138, 139).

Doughs meant for freezing are usually prepared at lower temperatures to limit prefreezing fermentation (140), with best results achieved with mixing temperatures around 20°C (136). A compromise freezing rate is needed to freeze the dough, slow enough to maximize yeast activity but fast enough to limit dough weakening. Freezing rates of 0.3 to 1.2°C/min have been recommended (128). Air-blast and contact freezers have been used successfully for freezing dough.

Baked bread can also be frozen, and studies have shown that freezing can retard staling (142, 143). However, as retrogradation proceeds faster at temperatures between  $-3^{\circ}$ C and  $5^{\circ}$ C, it is important to cool and freeze the bread rapidly (144). Cryogenic freezing is an excellent means for freezing bread, but air-blast freezing can also be effective (145). Avoiding dead spaces in the wrapping helps enhance the rate of freezing.

Moisture loss during frozen storage can result in a dry, firm texture (146, 147). The useful storage life of frozen bread is often limited by the pickup of undesirable flavors. Storage temperatures should be less than  $-18^{\circ}$ C to prevent retrogradation during storage (147). As with other frozen foods, temperature fluctuations in frozen storage are particularly detrimental to textural quality. Proper thawing of bread is also important to

limit staling, firming, and loss of moisture (148). Breads rapidly thawed at 40–60% relative humidity have the fewest changes in texture (149).

#### IX. CONCLUSIONS

Due to the chemical and structural differences in different food groups, each has unique issues associated with changes in textural quality. With the exception of bread dough, most food groups suffer fewest changes in textural quality when frozen at a rapid rate. In addition, storage at low temperatures is preferential, and particular care should be taken to limit temperature fluctuations during frozen storage. Methods of thawing can also affect the texture of foods. However, the variety of thawing regimes is limited, particularly as much thawing is accomplished by consumers.

This chapter has reviewed the effects and processes of freezing major food groups. In addition to these, there are a number of further-processed entrees and heterogeneous foods that are frozen and prepared as convenience meals. Although the issues associated with these foods are too numerous to cover here, it is hoped that this chapter can also be used as a starting guide for understanding such foods.

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## 9

# Frozen Muscle Foods: Principles, Quality, and Shelf Life

Natalia F. González-Méndez, José Felipe Alemán-Escobedo, Libertad Zamorano-García, and Juan Pedro Camou-Arriola

Centro de Investigación en Alimentación y Desarrollo, A.C., Hermosillo, Sonora, Mexico

#### I. INTRODUCTION

Worldwide population growth, the increasing pace of the modern lifestyle, the changes in the role of women, and the rise in the numbers of people living alone in the major developed countries have resulted in a constant increase in the use of frozen vegetable and animal food products. Frozen foods mean an increased shelf life and a quality of preserved food comparable to that of fresh food.

Freezing perishable food such as poultry and meat seems to be an easy operation. At first sight, it looks like a mere temperature decrease until ice is formed in the food (1,2). However, the freezing that matters to us does not happen in this way, since once a process is carried out, internal changes that affect quality and shelf life take place. This has been very closely studied by researchers for decades, in order to know its advantages and disadvantages as a conservation method and the problems related to the process itself.

Knowledge of the solidification process produced during freezing has permitted wide and assorted applications, including genetic conservation material (germoplasm), damage by frozen plants and soils, frozen organs for medical application and, of course, a wide range of food conservation.

Freezing is a unitary operation in which a food's temperature is reduced below the freezing point and a proportion of water experiences a change to ice crystals. This crystallization lowers water activity and removes water from food. Shelf life is increased, and its deterioration by enzymatic, physical, chemical, and microbiological changes is avoided. Three different stages are distinguished in this conservation method practice: (a) food freezing, (b) frozen product storage, and (c) thawing (3, 5). Freezing accomplishes the first two stages, a simple temperature reduction followed by storage at a freezing temperature of under  $-18^{\circ}$ C. Food thawing, is a critical stage affecting the product's final quality, and it is often ignored and/or misunderstood. This chapter focuses on the principles of freezing and accompanying factors that affect the quality and shelf life of poultry and meat products during the three stages.

#### **II. FREEZING PRINCIPLES**

As it was previously mentioned, freezing is a unitary operation that involves food temperature reduction and change of state from water into a solid (ice). Both actions are responsible for the conservation power of this method. The term freezing is usually applied to this process, and particularly until a food temperature descends to  $-18^{\circ}$ C (0°F) (6). Obviously, this water state change within food involves physical aspects and physicochemical changes that reverse its importance in quality and shelf life from a frozen product. Poultry and meat products in this matter are not exceptions, as will be seen below.

#### A. Physical Aspects

Great size refrigeration machinery is required in order to remove heat and to decrease product temperature to accomplish industrial freezing. This is the so called freezing process, but one must underline that heat removal itself is not freezing; it is part of the process (2). This leads us to indicate that the method goes through three physical states so a food can actually freeze and stay in such a state (6-8): (a) cooling a product toward freezing temperature range (freezing point), (b) product freezing, and (c) cooling the product at storage temperature. The first product-cooling step is obligatory and it does not necessarily reach the frozen state. This is achieved by eliminating sensitive heat from the system, which is the only factor that can be removed without reaching the frozen state (temperature differential dependent step from the center to the surface, known as "cold front"). In order to reach crystallization one must also extract the latent heat (modifying system enthalpy) to produce supercooling. At this point, nucleation takes place with nuclei or seed formation that provokes a temperature increase due to released crystallization latent heat. At this point it can be said that a food freezing phase has begun. Crystallization is determined by two factors: (a) nuclei formation which occurs from -1 to  $-7^{\circ}$ C in a homogeneous or heterogeneous way, and (b) crystal growth speed, which appears once crystallization nuclei have been formed. Cooling speed influences the quantity and form of the generated crystals. Three main types of crystals distinguished are hexagonal regular crystals, irregular dendrites, and spherical crystals.

Furthermore, a large ice crystal formation or recrystallization can take place, followed by a continuous diffusion of water vapor. Large crystal ice formation and temperature oscillations have a negative influence on the quality of a frozen product (3, 9).

Studies of poultry, pork, and bovine meat freezing and the effect of speed and type of crystals is contradictory (10–12). In most of them, the mechanism and freezing effect on water loss is determined, and only a few present histology studies show the ultrastructure and possible damage occurring at different freezing speeds. The greatest difficulty faced by these studies is the great variability of meat material before freezing (nature, composition, tissue distribution that compose it, handling history, etc.). This preservation method, like any other, *does not improve the quality* of meats, so it is quite important to make a food selection very carefully before freezing it.

#### B. Freezing Rate

The freezing speed affects meat's physical (13) and chemical properties, and it is influenced by the amount of fat present that has a smaller thermal capacity than lean tissue. Since

freezing speed is increased by water, its efficiency can only be improved by increasing the relative humidity (7). There are two types of freezing speeds:

1. Slow Freezing. This is a process in which the temperature of a product that will be frozen remains next to an initial freezing point for a considerable period of time. This causes a continuous freezing frontier to be formed that slowly progresses from exterior to interior of a product. Extracellular water is frozen faster than the intracellular water because it has a lesser solute concentration. This favors ice crystal formation and a solute concentration in a nonfrozen solution. Furthermore, intracellular nucleation behavior can be deficient owing to the higher solute concentration in the sarcoplasm.

Mechanical damage occurs easier in this freezing method owing to an expansion associated with excessive frost accumulation formation, as well as with a contraction from the muscular fibers that have lost water. The muscle under these conditions presents a distortion of its normal aspect (3, 14–16).

2. Rapid freezing. A more rapid freezing speed is obtained when condensed gases such as liquid nitrogen, solid carbon dioxide, or liquid nitrous oxide are employed. Temperature from meat products that are going to be frozen falls quickly below initial freezing point, forming numerous uniform ice crystals over all the meat tissue. This has a filamentous effect, spreading from muscle fibers to the interior and exterior at about the same speed. Owing to a rapid temperature fall, the ice crystals formed have little possibility of increasing their size. Therefore a rapid freezing determines the spontaneous formation of small and individual ice crystals. As a result, a rapid meat freezing causes less detrimental effects than slow freezing (3, 7, 14, 16, 17).

#### C. Freezing Time

As in freezing rate, freezing time is a very important parameter from the point of view of design, application, operation, and process handling. This knowledge permits us to determine or predict process requirements and economical aspects, including its impact on product quality, which depends on product's latent heat of fusion removal velocity and small ice crystal production. (18–21). Small ice crystal formation is determined by heat removal speed. Product heat transfer is accomplished by conduction from the interior to the surface, and from there to freezing medium. Heat transfer velocity is determined by (6, 22): (a) initial and final temperatures; (b) product thermal conductivity; (c) exposed food area (product form and dimension, particularly its thickness); (d) temperature difference between food and freezing medium; (e) insulation effect by the air that surrounds the product; (f) enthalpy variation; and (g) product and package heat transfer coefficient.

According to the literature, freezing time is the time elapsed from the start of the prefreezing stage until the final temperature is reached (8). Nevertheless, some authors do not agree with this definition, since product freezing point has to be defined first (5). This is because foods freeze over a range of temperatures. Temperature distribution is a variable that depends upon where the temperature is monitored. This internal temperature variation shows a thermal center, which is referred to as the area where the slowest cooling occurs, and generally is used as a reference for predicting or calculating freezing time. Two concepts have been established: the "effective freezing time" and the "nominal freezing time" (NFT) (6, 15, 22). The first is the time required to lower the temperature of a food at its thermal center to a desired temperature below the initial freezing point, and is used as a measure for frozen time storage. The second concept is the time between when the food

surface reaches 0°C and the thermal center 10°C below its freezing temperature. This is used to determine food deterioration.

Freezing time calculation is not an easy task, since it involves a complex problem of heat transfer along with phase change, variable thermal properties, and most of the time anisotropy problems. This is provoked by product form and size differences, initial product temperature, freezing temperatures, different crystallization speeds within a food, and changes that occur in density, thermal conductivity, specific heat, and heat transfer during product cooling (20, 22, 23). For this reason, freezing time calculation can be done experimentally by observing product temperatures (in equilibrium) and solving by analytical or numerical mathematical methods. The first method involves relatively simple equations, and they are the most used. The numerical models require the use of a computer, and are more precise (15, 21, 23). Since 1941, the analytical models are based on the Planck equation and its posterior modification (6, 21, 23). The assumptions and boundary conditions and its accuracy depend on how closely the corresponding assumptions approach reality (6, 7, 18, 19, 22–31). More than 30 mathematical methods to predict freezing time have been reviewed and published (7, 19, 22, 23).

#### D. Physicochemical Changes

Among the most important physicochemical changes that occur in this type of foods that affect quality and functionality of meat proteins are the following:

The first change occurs while free water present in poultry and meat products becomes ice. During this solidification an osmotic type of effect takes place as solids keep concentrating in a progressive way (salt or chemicals added) resulting in cellular damage of meat or in structural proteins (mainly myosin) in processed products (22, 32–35).

Another physicochemical change that may cause cellular damage is the growth of ice crystals; tissue disorganization or structure deterioration occur according to the rate of freezing (large or small ice crystals) (32, 34, 35).

Another change happens when ice present in the product periphery is sublimated, carrying on a superficial dehydration that affects product appearance and favors oxidation reactions. This sublimation is called freezer burn and is manifested by a brown stain in red meat products and by superficial dehydration or discoloration on poultry skin. Fats also experience a coloration change (to a gray color) by lipolisis action; furthermore, free fatty acid oxidation confers a yellow color (1,3,35). Water is not totally reabsorbed by the product (shrinkage), and as temperature variations continue, excessive frost accumulation can represent losses (1, 34). Sarcoplasmic protein loss is caused by this and by a decrease in collagen solubility that affects hardness in meat. When a loss of juiciness is apparent, it reflects a water retention capacity loss (CRA) caused by protein denaturation during storage (32, 36). Studies in beef and pork showed that long freezing and storage periods affect texture and tenderness. This could be a consequence of a wrong freezing method for those particular cases (37, 38). Shrinkage and freezer burn also affect fresh ham and results in reduced prices (34). Nevertheless, the rate of freezing and thawing shows the influence on the salt diffusion of beef and pork. This offers an advantage in the curing process (3, 39).

#### E. Methods, Storage, and Thawing

Industrial or home equipment used to freeze foods is related to the method used. Final product quality and shelf life will be affected by the method employed; therefore a careful

method selection is required. In addition to equipment cost, energy is needed to support temperature decreases. The nature and quality of the meat should also be considered before freezing it. It has been reported that the best way to preserve quality in special cuts or whole poultry is by rapid freezing owing to the high content of unsaturated lipids in the products. Red meats, with the exception of ovinecaprine and meat products such as ham, bacon, and sausage, do not fit this method.

#### 1. Methods

At present, a vast range of technologies (equipment and processes), which are versatile and facilitate an adequate freezing for each kind of meat and poultry product, is available. The methods can be classified as: (a) contact with cold solids, (b) contact with cooled liquids, (c) contact with a cooled gas, and (d) cryogenic freezers (two phases). These methods, as well as the required industrial equipment to apply them, are described in Chapter 11. High pressure along with freezing has recently been used to improve final quality (40). This method is considered a new technology.

#### 2. Storage

The conditions under which frozen meat is stored and distributed (containers for its transportation, showcases for retail sales, etc.) can become crucial for poultry and meat product quality and shelf life maintenance. An adequate meat storage period varies according to the kind and the type of product. Furthermore, it is influenced by freezer temperature, temperature fluctuations, and packaging material quality (41).

Shelf life for all different types of frozen meat can be extended by reducing storage temperature. Most chemical changes can be eliminated by decreasing temperature to  $-80^{\circ}$ C, but such temperatures are not economically feasible in most stores. The growth of microorganisms that cause putrefaction and deterioration, and most enzymatic reactions, are quite reduced at temperatures inferior to  $-10^{\circ}$ C. As a rule of thumb, industrial freezer units handle temperatures lower than  $-18^{\circ}$ C (0°F).

Metabolic activity from all pathogenic bacteria is paralyzed at temperatures below  $3^{\circ}$ C. Temperatures below  $-10^{\circ}$ C inhibit bacteria growth completely and most of yeasts and molds. Therefore, storage at  $-18^{\circ}$ C can be considered as a good control method of microbial flora (42). Only bacteria spores (specially Bacillus and Clostridium, like Cl. Botulinum) resist freezing storage temperatures (22).

#### 3. Thawing

Meat quality depends on: condition after slaughter, freezing technique, storage climatic conditions, and proper thawing. Most advantages associated with a correct thawing method disappear when it is not done properly (17). A greater amount of cellular water reabsorption is reached when carcass thawing is slow. In fast carcass thawing more meat juice is expelled. Valuable flavor and mineral substances are lost along with it; therefore a rapid thawed meat is substandard and drier (3, 14).

There are two different types of thawing methods. The first consists of providing heat to a product surface by exposing it to air, steam, other liquids, or hot surfaces. In the second procedure, the heat is generated within a product by microwave warming or electricity, or by taking advantage of the electrical resistance. Research on different freezing applications is focused on damage levels suffered by the meat in its structure, functionality, and quality characteristics.

It was found by Barbut and Mittal (43) that poultry was suffering damage in its structure due to freezing, showing meaningful losses in water holding capacity (WHC) and a decrease in its functional properties and quality as a consequence. Abdel-Gawwad et al. (44) found that, in addition to protein loss, the quantity of nutrients such as amino acids, iron, and vitamin B in buffalo meat is also reduced by the liquid lost upon reducing the WHC (44). They applied a low freezing method, and the meat was kept in good conditions for 3 months. In a study by Farouk and Swam (45), different functional properties and quality meat characteristics were correlated. Their experiments showed that the negative changes in beef can be altered (during freezing) if time and rigor temperature are modified.

#### III. QUALITY AND SHELF LIFE

Deterioration processes are temperature dependent, especially in frozen poultry and meat products. The rate of deterioration in quality and shelf life is slowed as the product is exposed to a lower temperature. However, this factor is not the only one that provokes changes in frozen product quality and shelf life. Other important factors are (a) product nature and its quality before freezing, (b) handling and previous operations before and at freezing, (c) packaging, (d) temperature fluctuation (range or tolerance), and (e) storage time. The first three factors are known as product—processing—packaging (PPP) and the last two are described as time—temperature—tolerance (TTT) (6,8). The sanitation of poultry and meat products during handling can suffer adversely, especially during slow freezing. Thus, since freezing does not improve product quality, and fluctuations existing during freezing storage are not controlled, it can deteriorate even more.

Quality loss in poultry and meat products caused by chemical changes and, in some cases, by enzymatic activity during frozen storage  $(-18^{\circ}\text{C})$  are slow, but they are accelerated by the effect of solids concentration around the ice crystals as a result of reduction in water activity, pH, and ox-redox potential (22, 32).

Although the freezing process is one of the most important methods of prolonging the shelf life of meat and poultry, deterioration in quality characteristics (texture, flavor, and color) resulting from protein denaturation (biochemical and physicochemical changes) may arise. Muscle proteins are susceptible to freeze denaturation, losing their functional properties (water-holding ability, viscosity, gelation, emulsification, foaming, and whipping). Another factor involved in this denaturation and deterioration is lipid peroxidation during frozen storage (32, 34, 35).

#### IV. CONCLUSIONS

Although the deterioration of poultry and meat products due to freezing has been established, freezing remains one of the best methods that will permit the preservation of physical and organoleptic characteristics close to those of the products before freezing. This offers great use in large cities and developed countries. PPP and TTT observation along with good hygiene during manufacturing operations (good manufacturing practices) permit a control over quality and shelf life of these frozen foods, providing more time for their distribution and consumption. On the other hand, much negative discussion on freezing, storage and defrosting effects has been published. Also, scientific literature on

thawing effects of frozen foods is limited. In brief, the proper selection of a right freezing method depends directly on what the consumers prefer for a frozen product and its storage period.

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# 10

# **Operational Processes for Frozen Red Meat**

M. R. Rosmini

Universidad Nacional del Litoral, Santa Fe, Argentina

J. A. Pérez-Alvarez and J. Fernández-López

Miguel Hernandez University, Orihuela, Spain

## I. INTRODUCTION

Since ancient times, humanity has known the advantages of low temperature as a food preservation method. Certainly this knowledge was acquired through observing the effect that low environmental temperature had on vegetables and meat stored for food.

The development of mechanical refrigeration and the granting of the first patents for freezing food during the 19th century is the starting point for the use of commercial freezing.

In the beginning, the use of freezing as a meat preservation method allowed the commercial exchange of frozen carcasses or quarters among European consumption centers and production centers located in Oceania and the Americas. The lack of frozen storage systems at homes was the cause of restricted production and commercialization of small frozen cuts intended for domestic use. By the second half of the 20th century there was a significant increase of freezers in Western homes. This fact led to the development of a great variety of frozen foods in general and of meats in particular.

The initial notorious reputation that frozen meat had owing to quality loss by cold negative effects (burns, texture and color changes, etc.) had progressively disappeared by the mid-20th century, especially owing to the development of efficient technological processes causing relatively few modifications of organoleptic features.

The order that muscle cell structure has shows that meat is an organized biological system. Upon completion of industrial slaughtering, a great number of natural defensive barriers (immune system, pelt coverage, structure integrity, etc.) that prevent microorganisms from invading tissues during life are inhibited. The lack of such defensive systems, added to the loss of tissue internal balance, makes meat one of the most perishable foods.

Freezing is an excellent procedure to keep the original characteristics of fresh meat almost intact (nutritive, organoleptic, and sanitary features) for long periods (1). The efficiency of this method is based on two phenomena that take place in the tissue: (a) temperature decrease, which slows tissue and microorganisms' own chemical reactions; (b) internal dehydration owing to ice crystal formation that decreases water activity.

On the other hand, meat freezing allows coping with market fluctuations, thus regulating prices through meeting the needs of supply and demand; it satisfies international commerce, especially reaching those markets that are far from production locations and, it eases urban living conditions. People employ less time in purchasing and preparing meals and demand more ready-to-cook or ready-to-serve food.

#### II. BIOCHEMICAL CONSIDERATIONS

Food is considered to be frozen when most parts of its freezable water component is ice. The quantity of ice formation depends on temperature and food composition (2). In meat, this phenomenon is progressive and fast up to  $-5^{\circ}$ C, freezing approximately 75% of tissue fluids when such temperature is reached, 80% at  $-7^{\circ}$ C; 90% at  $-8^{\circ}$ C, and 98% at  $-20^{\circ}$ C. Complete crystal formation is achieved at  $-65^{\circ}$ C. Nevertheless, more than 10% of the water that composes muscle will not freeze owing to the strong union it has with proteins.

In contrast to what happens to fresh muscle, frozen meat may show modification due to cell structural damage and disfunction of certain cell systems (3). The freezeable portion of tissue fluids (free water) contain a great amount of organic and inorganic solutes that are responsible for decreasing meat freezing point from 0 to  $-1.5^{\circ}$ C.

As temperature decreases, crystal formation takes place (nucleation) (1). Such crystals are distributed among intra- and extracellular spaces according to the time necessary to go through a temperature range from -1 to  $-7^{\circ}$ C (4,5). When this period is greater than 2 hours (slow freezing), crystals form outside the cell, and extracellular solutes concentrate (1). This causes the release of intracellular fluid to compensate for the osmotic imbalance, but the new fluid freezes, causing an increase in size of extracellular crystals and consequently higher pressures on muscle fibers owing to an enlargement in volume. This increase is about 9% of pure water (4). The increase of crystal size takes place at a constant temperature. Temperature fluctuations accelerate the process (6).

The slower the freezing process, the greater its denaturant effect on myofibrillar proteins and cow muscle (7). An extended exposure to adverse pH values and high saline concentrations causes the denaturation of protein, the subsequent decrease in the ability to hold water (8), and a high fluid dripping at meat thawing. The mechanical damage caused by big crystals on muscle fibers contributes to this phenomenon (9).

On the contrary, when this period is shorter than 2 hours (rapid freezing), water will freeze in its original location, and small crystals will be formed not only within but also outside the cell (1). When freezing is carried out rapidly and by means of low temperature, the water holding capacity of meat is greater. Therefore big crystal formation and myofibrillar protein disruption is lower (10, 11). Thus freezing speed will directly influence meat quality (12).

Apart from the velocity of freezing, meat quality is affected by the typical characteristics of the raw material (intrinsic quality), the time it remained refrigerated before freezing, and the storage period and conditions of storage (temperature, humidity, packing material) (8, 11–14).

If raw material handling is adequate during freezing and storage, most of the nutritive value will be preserved, as the loss of nutrients (salts, amino acids, proteins, peptides, and water-soluble vitamins) is due to dripping or exudation at thaw. There is practically no nutrient destruction or loss of digestibility in comparison with other preservation methods.

Nevertheless, cold treatment does not completely stop physical, chemical, or biochemical reactions that take place in animal meat after slaughter, which means that frozen meat has a limited preservation capacity. Fat alteration processes (oxidation, rancidity), and decoloring are instances of limiting factors to be considered when preserving meat by freezing (15, 16) (see also Chapters 10–15).

#### III. MEAT FREEZING

Specific demands from different consumer markets can be seen in the wide variety of commercial agreements that companies have to fulfil by means of the utilization of the whole carcass.

The existence of different methods for preparing meat before freezing will be determined by demand changes regarding the various markets, local, regional, or international, each company's own commercial relationships, and specifically the final use of meat.

These methodologies may represent more or less complex processes of preparing and handling meat. Such processes comprise industrial cattle slaughtering and carcass freezing, primary divisions of carcasses in order to freeze quarters and ribs, deboning big muscle cuts to preserve complete anatomic regions, cleaning and preparing special commercial cuts, prepararing weight-controlled portions, and even mincing and formulating new products that will be frozen further.

In turn, the food industry has different freezing methods that can be applied to product freezing: air blast freezing, contact freezing, immersion freezing, and cryogenic freezing. Even though all of them may be generally applied to the meat industry, the system applied in each process line will depend on the specific needs of production and the feasibility of adjusting the system to other line operations (preparation, processing, packing, etc.) (17).

### A. Carcass Freezing

Carcasses are the end products of the industrial cattle slaughtering process intended for human consumption. The meat to be preserved by freezing should come from healthy animals, slaughtered under strict sanitary conditions. The best carcasses are those coming from well-nourished animals, with an appropriate muscular formation and an external fat coverage that will protect them from the damage caused by cold.

Carcasses, as well as front- and hindquarters and ribs, may be frozen without protective packing. As piece size decreases and fat is removed, such protection becomes necessary to avoid the damages of freezing. Frozen meat stored without packing for a long period may show surface damage due to tissue water loss through sublimation. The results of this deterioration, which takes place especially when freezing rooms have low humidity and high air movement, is known as freezing burns.

To reach an adequate preservation quality, the carcass freezing process should be carried out immediately after slaughtering or subsequent refrigeration, as part of the process on the same premises. This will prevent the development of the microorganisms present and will delay the effects of the natural enzymatic system. A well integrated process allows good efficiency levels by means of a good use of energy and raw material, avoiding handling related to loading, transportation, and unloading, which are necessary to carry carcasses from one company to another.

The carcass freezing process may be carried out with or without a prior refrigeration stage. Thus a direct single-stage freezing, without prior refrigeration, will be applied after slaughtering, whereas a two-stage freezing will be carried out with a prior carcass refrigerating process.

The advantages of the first system are that it allows a shorter total freezing time (within 8 to 15 hours according to animal species and the process used), it needs less carcass handling, and it decreases the natural weight losses that take place during refrigeration in the two-stage system. Another advantage is related to the immediate interruption of microorganism multiplication by applying low temperatures and consequently quality improvement in the preservation capacity of single-stage frozen meat.

Besides, carcass freezing without prior refrigeration, i.e., before rigor mortis appears, does not allow meat to undergo its normal maturation process. Rigor mortis will occur at thaw and will result in texture deterioration. This phenomenon is a disadvantage and could be lessened in different ways: using high-speed freezing (difficult to apply in a carcass), extending thawing time in order to complete maturation and make rigor mortis disappear, or applying an electrical stimulation during animal slaughtering (10, 18–20).

It is also possible to shorten thawing time with two-stage freezing, using sudden refrigeration. This has negative consequences on texture and water holding capacity. In such cases it is convenient to regulate cooling in such a way that any part of the carcass reaches 10°C before 10 hours.

Generally, the carcass or quarter freezing process is carried out by applying air blast freezing in tunnels or cold storage rooms. Pieces are hung along rails, transport tracks, or room framework or tunnels, in such a way that they do not have contact with one another, or with the building structures. Refrigerated air should flow without barriers in order to guarantee all pieces are reached with the least possible resistance. The piece should be loaded taking into account its technical characteristics. An overload will prolong freezing time and will decrease industrial output. Once the carcasses are placed, they are subjected to temperatures of -25 to  $-40^{\circ}$ C and an air speed of 2 to  $4 \, \text{m/s}$ . This process allows internal muscle temperature to reach -18 to  $-21^{\circ}$ C within 48-72 hours. At a first stage, freezing process velocity will depend upon circulating air flow. The higher the speed, the greater the heat exchanges between carcass surface and circulating air. Later, once the carcass surface has reached freezing temperature, there will be a greater dependency on air temperature and meat thermal conductivity.

Freezing alters carcass mechanical characteristics: it makes them stiff and allows them to be piled up on trays provided that the cold air reaches all pieces and that in no case they have direct contact with the floor.

The pieces leave the tunnel or chilling room once they reach the final temperature under which they will be stored. In order to receive the product, these cold storage rooms will be clean and free from abnormal odors. Storing condition fluctuations should be avoided. Special attention should be given to the fact that storing fresh and frozen meat together affects the final quality of the product (6). Greater damage in meat takes place when temperature variations occur below  $-12^{\circ}$ C; above this value, and especially from  $-18^{\circ}$ C, the effects are less harmful. In case carcasses from the same lot show remarkable differences in size it is sensible to locate the biggest pieces where the freezing conditions are best.

The freezing storage period as well as the final quality of the product will depend on many factors: (a) characteristics of the raw material (animal species, carcass size, fat degree, initial temperature); (b) the slaughtering process conditions and procedures used during its development; (c) the freezing process conditions (refrigerant evaporation temperature, air rate in the tunnel, type and density of the load); and (d) surface desiccation of meat and lipid rancidity.

Preservation of meat subject to temperatures of -18 to  $-21^{\circ}$ C and 90% humidity is effective for 15 months (10).

# B. Freezing of Meat Cuts

The preparation and freezing of cuts of meat have many potential advantages in comparison to fresh meat commercialization, especially when it comes to supplying distant markets with the most profitable cuts only.

On the other hand, frozen meat cuts, in contrast with frozen carcasses, have economic and technological advantages that justifiy a massive practice: meat cuts preparation and packing can be done at the same slaughtering plant. This allows access to mass production economies, freezing only those cuts of high commercial value, superior quality, and specific market demand. It also avoids the freezing of bone structures (approximately 20% carcass weight). A more efficient processing of subproducts can be achieved. It doubles the quantity of meat per units, which in turn can be seen as a better use of cold areas from the industrial stage (tunnels, cold rooms, storage rooms and transportation) to the commercial stage (exhibition freezers). It decreases costs of transportation to consumers. It allows meat packing with which weight loss and surface quality deterioration due to cold is reduced. Also, sanitary handling of meat during distribution and commercialization stages becomes easier.

In some countries, producing frozen meat cuts can be a way of supplying government supported institutions (homes, day care centers, schools, prisons, etc.), as well as restaurants. This improves catering and other aspects making a better use of resources.

Good carcasses and quarters with an adequate distribution of fat coming from healthy animals slaughtered under aproppriate sanitary procedures are used in the preparation of frozen cuts. It is advisable for refrigerated meat not to have undergone a maturation period of over 24 hours, because as time passes, undesirable modifications increase during frozen storage.

Production begins with carving a carcass to obtain ribs and quarters. This operation should be carried out under sanitary conditions in a room with an ambient temperature not above 10°C.

The large muscle masses are obtained by carving the quarters manually with a knife. Disjoining these anatomic regions (Fig. 1), meat cuts surrounded by fascia, aponeuroses, nerves, and vascular groups are obtained. Pieces are prepared according to the specifications of each country, in general, and to purchaser requirements in particular. Undesired tissues are removed and surfaces are trimmed so as to obtain a neat aspect.

As end products resulting from the preparation process, different meat cuts are obtained (Fig. 2). Those of a higher commercial value and better quality are selected to be frozen. A varying amount of cuts (lean and fat) results in a byproduct that can be used as raw material—fresh or frozen—in the manufacturing of meat products without anatomical integrity.

The prepared meat pieces are protected individually by means of primary packing. When packing is carried out in a normal atmosphere, plastic foil that closely attaches to meat is used. It is important not to leave empty spaces between the wrapping and the meat so as to avoid weight loss due to ice sublimation. In the case of vacuum packing, pieces are placed into bags made of contractile plastic layers. The air is removed, the bag is sealed



Figure 1 Cuts and trimmings are obtained from big muscle regions in a refrigerated room.

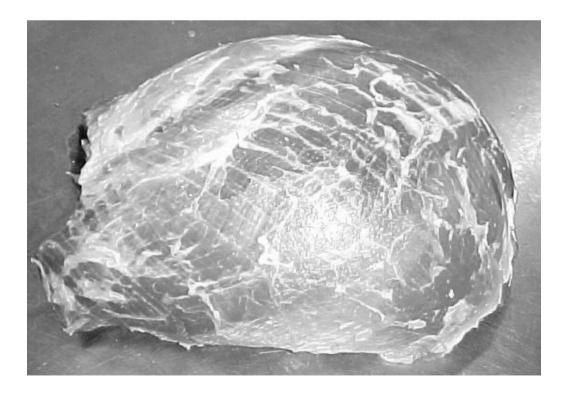


Figure 2 Deboned beef cut ready to freeze.

thermally and immersed for a few seconds into lukewarm water bath or conveyed into a hot air tunnel to favor contraction of the plastic wrap onto the meat. Bags should be of a low permeability to water vapor and gases.

Primary packaging should achieve the following goals, some major, some minor.

- 1. Product protection
- 2. Resistance to changes in temperature and humidity and to mechanical impact
- 3. As a barrier to odors, tastes, and poisonous substances
- 4. Adaptability to shape of product
- 5. Flexibility in freezing temperatures
- 6. Competitiveness in price

Among the acceptable materials include polyethylene and polyvinyl chloride.

Once the cuts are wrapped or in plastic bags they are placed in folding pasteboard boxes with a cover that will serve as a protection during storage and distribution. Secondary packing should be light  $(250 \, \text{g/m}^2)$  and resistant to breakage, poking, and traction. This quality is determined by wall thickness (3 to 11 mm).

Boxes, are approximately 600 mm long, 400 mm wide, and 150 mm high. They can hold a varying number of cuts according to their type and size. A box can weigh approximately 27 kg. The pieces should be arranged in a single layer so as to favor cold penetration during the freezing period.

The freezing process of the cuts should be carried out fast. The freezing rate of the piece must range from 1 to 5 cm/h, to minimize structural damage and immediately stop the negative effects of microorganisms and natural enzymatic systems.

The boxes can be placed in high-rate air blast tunnels at an ambient temperature of  $-40^{\circ}$ C. In order to favor cold penetration and accelerate the freezing process, the boxes are placed on shelves. There should be some space left between boxes so that there is a greater surface for cold penetration. The cold airflow can be oriented in a parallel or perpendicular direction to the boxes according to the disposition of fans along the freezing tunnel. This system is effective and economical, but the heat transfer coefficient is low compared to contact systems.

In some cold storage rooms, the flat surfaces of the shelves on which boxes are placed are made of tubes. A liquid refrigerant circulates inside the tubes. In such cases there is only direct contact with the botton of the box. Sometimes, to speed the freezing process, fans force cold air to circulate at a high rate.

On the other hand, plate freezers have a hydraulic or pneumatic approximation device that applies a slight pressure onto the boxes that allows contact at the top and the bottom of the boxes. Everything is enclosed in a chamber, which allows a better use of cold. Thus the system is more efficient by increasing the surface of contact and the heat transfer rate and by decreasing the surface devoted to the process.

The freezing rate in contact systems is higher than in the air blast system. Whereas in the former, meat can reach  $-18^{\circ}$ C within 14 to 16 h, in the latter, 20 to 24 h are necessary to reach the same temperature.

Once the meat is frozen, the boxes are stored in rooms with an ambient temperature of  $-30^{\circ}$ C. To make an effective use of the space, boxes are piled up on plastic or wooden pallets to avoid contact with the floor and to facilitate transport by means of a forklift. The base of the pile is two boxes by three. The following layers are interlocked to render the pile safe. The pile should not be higher than ten boxes.

The natural characteristics of meat remain intact for 12 months if there are no temperature fluctuations and the container is not altered.

# C. Weight Controlled Portion Freezing

The private restaurants and hotels as well as catering services consider the frozen weight control portion to be an advantageous product from the operative and commertial point of view. By the use of portions they can adjust to a varying demand, they can diversify what they offer, improve the cooking process, and increase benefits by a strict calculation of their needs.

These portions are also offered at supermarkets to target a segment of the population that, without consuming meat massively, demands a high quality level and a packed product that is easy to prepare and ready to eat. Besides, portions are ideal for supplying thinly populated areas, which can be reached through a distribution system that guarantees the cold chain.

Portions should be prepared in refrigerated rooms (maximum ambient temperature: 10°C). The process should be fast, so that the cold loss in meat is at a minimum.

The production process can be performed in two ways: starting from deboned frozen meat pieces or from refrigerated deboned pieces.

In the first case the process begins from a deboned frozen meat cut, the temperature of which ranges from -18 to  $-21^{\circ}$ C, meeting the needs of a specific market, for example, ribeye roll steak is obtained from beef rib and strip loin steak is obtained from beef loin (Fig. 3).

The primary packing is removed from the prepared and frozen cut, which is molded by means of a press and then compacted and shaped. Later, the piece is carved into portions. The machine that cuts the meat can be adjusted for the width of cut. The blade cuts the fiber perpendicularly, taking into account that wider pieces are heavier. Each unit is weighed individually to verify that it is within the tolerance limits agreed upon with the buyer (ranging from 150 to 500 g, according to future use).

Each portion is vacuum-packed individually, using contractile plastic that is cold resistant and impermeable to gas and humidity. It should block all kinds of exchanges with the exterior atmosphere (primary packing).

Secondary packing is prepared with a varying number of pieces according to its commercial aim. If the pack is designed for a supermarket, two to ten pieces are placed in a cardboard container of medium resistance. This is the pack to be handled by the consumer. Many of these containers are placed in a high-resistance cardboard box to provide greater protection during storage and distribution. When they are aimed at massive consumption services (restaurants, hotels, etc.), a bigger number of pieces are placed in a carton.

In the second case, the portions are carved from a refrigerated and matured piece, and it is necessary to consider a certain overweight (2–3%) in relation to the final value to compensate for the possible loss during manipulation. The pieces are packed individually as described above. They are placed in the secondary containers and are frozen in air blast tunnels or plate freezers. This system is suitable for portions prepared from pieces of a round shape, for example a beef round top, from which a top round steak is obtained.

Finally, high-resistance cartons are stored in rooms having a temperature of  $-30^{\circ}$ C.

## D. Meat Trimming Freezing

As a result of meat cut preparation there is a great amount of trimmings (lean and fat) of a variable shape and size (Fig. 1), which can be used fresh or frozen, in the manufacture of meat products.



Figure 3 Beef loin.

A simple process is performed for their preparation. The trimmings are clasified according to their proportion of lean and fat to obtain meat of different quality aimed at different industrial processes. Trimmings are placed in plastic bags (primary packing) and later in cardboard boxes (secondary packing) so that they can become a trimming block when frozen (Fig. 4). These blocks weigh 12 to 25 kg for easy handling. In some cases, when the blocks are to be used in the same factory, the boxes are replaced by reusable metal trays, which serve as molds and are coated by a plastic film (Fig. 5). This makes the process cheaper because of the absence of the secondary cardboard packing. The boxes and trays are placed in freezing tunnels or plate freezer systems (Fig. 6). The freezing rate will vary according to block size and the device used. Whereas in the plate system 18 hours are necessary for the 25 kg blocks to reach freezing, in the air blast system the period is extended to 24 hours.

Blocks are an efficient and safe form of storing meat for industrial uses. Moreover, cuts of low commercial value or those that do not have a specific retailing demand can be



Figure 4 Frozen meat blocks.

more profitable if given an industrial use. Also, blocks are adequate in preserving seasonal excess meat to be reprocessed later.

Formation of salty and minced meat blocks is a variant to the process mentioned above. In this case, 2 or 3% salt is added to fresh trimmings, even those that are warm as



**Figure 5** Trimmings are shaped in reusable metal trays for freezing.

they come from deboning without prior refrigeration. Generally a cutter is used to ease size reduction, salt penetration, mixing and extraction of myofibrillar proteins. The meat paste obtained is placed in boxes or trays covered by a plastic foil and fast frozen at -21°C. Meat stored in this way has a good water-holding capacity and is an appropriate raw material for the manufacture of cooked meat products. Meat durability in storage is variable according to the species and packing conditions. Vacuum-packed beef maintains its quality from 4 to 6 months, whereas pork may show alterations in fats after 2 months.

# E. Cooked Meat Freezing

Frozen cooked meat is an interesting raw material for the food industry that prepares ready-to-serve plates. The cooking process gives an extra sanitation that, associated with the extension of its life span by means of freezing, makes the cooking process an adequate way of preserving meat raw material.

The manufacturing process begins with the carving of the quarters to obtain large muscle masses. From these anatomical regions are obtained the meat pieces with higher commercial value that will be devoted to the market that commercializes meat cuts. The remaining parts are trimmed with a knife to remove the "special" fat, fascias, and aponeurosis.

Meat chunks in the form of irregular cubes are variable in size according to the final use given to the product. When they are aimed at preparing meals with sauce and ground meat, the cubes weigh approximately 250 g. When they are used in the preparation of plates with medallions, they should weigh about 400 g.

Once the meat is minced, it is forced into plastic tubes of about 108 mm in diameter and 600 mm long, or 118 mm in diameter and 450 mm long. A combination of laminated plastic materials (polyethylene and polyamide) are used in the manufacture of these



Figure 6 Trays with trimming meat are frozen by plate freezer system.

containers to achieve heat resistance and let them maintain flexibility at freezing temperature. Once the tubes are stuffed, they weigh approximately 5.5 kg.

The tubes are sealed and immersed in water at 94°C for about  $2\frac{1}{2}$  hours to assure that the coldest part of the tube achieves 80°C. This temperature is considered to be enough to eliminate the majority of pathogenic bacteria—even the foot and mouth disease virus. Once the tube is removed from the cooking receptacle, it is drained to remove the broth. This juice, a by-product rich in proteins, mineral salts, and collagen, is an excellent raw source material to elaborate meat extract. The drained tube is compacted by forcing the meat into one of the two extremes. The interior pressure rises and the tube is stiff. Finally, a sealing clip can be put on the opposite extreme.

The tubes are placed in the plate freezing system or in shelves inside the air blast tunnel. Freezing is fast to lower the initial temperature of approximately 65 or  $70^{\circ}$ C to  $4^{\circ}$ C in the first 4 hours and  $-18^{\circ}$ C after 8 hours of freezing. The sudden decrease in temperature that meat undergoes during the first 4 hours is a significant thermal stress to the germs that may have survived the cooking process. Thus it increases the sanitary effect of such treatment.

The tubes of frozen meat are placed in cartons approximately  $680 \,\mathrm{mm}$  long,  $480 \,\mathrm{mm}$  wide, and  $120 \,\mathrm{mm}$  high (Fig. 7). There are 4 or 5 tubes in each box, according to their size. Then they are kept in storage rooms at  $-30^{\circ}\mathrm{C}$ .

# F. Minced Meat Freezing

In order to make the industrial process profitable, the meat industry must use 100% of products and by-products obtained from the animal. The carcass carving process and



**Figure 7** The tubes of cooked frozen meat are placed in cartons.

deboned-cuts manufacture produce a great number of meat trimmings that are unimportant to the consumer and that must be reduced in size to be useful for the industry.

Apart from this minced meat, there are a varying number of meat cuts that have no commercial value as anatomically identifiables pieces, so it is more profitable to use them as raw materials for making other products.

The mincing process is a mechanical operation that reduces the size of the meat particle. The rupture of tissue structure favors the exit of fluids that are not only inside muscle cell but also within intercellular spaces. It increases the surface of the meat exposed and destroys the last natural barriers the tissues have. This situation favors the development of microorganisms and the action of natural biological systems, making minced meat an extremely perishable product, thus justifying the use of cold as a preservation method.

There are different products made from minced meat that need to be frozen to extend their life span. Hamburgers, cutlets, and meatballs are among the products that are aimed at direct public consumption. Frozen minced meat blocks are an example of another industrial use of meat.

### 1. Products Aimed at Direct Consumption

The manufacturing of hamburgers and other products for direct consumption has the following process stages: mincing, mixing with additives and minority ingredients, shaping, freezing, and packing.

The shaping stage makes a great difference in several process lines requiring equipment with special molds for each necessary form: round for hamburgers, oval for cutlets, spherical for meat balls, etc. In all cases, the forming equipment has a dispenser that regulates the quantity of the mixture (meat and corresponding additives) to be introduced and a system generally formed by a mold that presses, compacts, and shapes the product. When the piece is a cutlet, the equipment has another system that dispenses liquid egg and breadcrumbs.

Once the product is shaped, it is placed onto a stainless steel continuous belt that carries it into an ascending tunnel in which cold air  $(-30^{\circ}\text{C})$  is forced to blow at high speed by means of fans. Product freezing will depend on the time inside the tunnel (conveyor speed), the number of units introduced, and the airflow rate.

During the shaping stage it is advisable to add excess weight of about 2–3%, which is lost in the freezing process as the pieces are introduced into the equipment without the primary packing. The weight of the end product is controlled either manually or automatically.

When the frozen pieces exit the tunnel, they go to the packing sector where they are placed into a plastic primary container and then into pasteboard boxes, or they are placed directly into cardboard boxes with a plastic or waxen coat inside. The packing varies the number of units according to the final use: 2 to 24 for domestic consumption and more than 30 units if they are aimed at restaurants, hotels, etc.

The product is kept in storage rooms at  $-30^{\circ}$ C; in domestic freezers the temperature should be at least  $-18^{\circ}$ C.

#### 2. Products Aimed at Industrialization

The process of preparing minced meat blocks has three different and defined stages: mincing, packing, and freezing. The size of the particle is variable, but generally nets with holes of 6 to 8 mm are used to allow for further industrialization.

The packing process involves putting bulk meat into plastic bags (primary packing) and then into cartons (secondary packing) that will give the shape of a block. The properties of the two packings and the dimensions of the boxes are similar to those described in the frozen meat cuts process.

Each block weights approximately 27 kg, and freezing may be performed in air blast tunnels or plate freezing systems.

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# 11

# **Frozen Meat: Processing Equipment**

Juan Pedro Camou-Arriola, Libertad Zamorano-García, Ana Guadalupe Luque-Alcaráz, and Natalia F. González-Méndez

Centro de Investigación en Alimentación y Desarrollo, A.C., Hermosillo, Sonora, Mexico

#### I. INTRODUCTION

Meat freezing is a necessity that has emerged and grown with the opening of world markets. The meat industry, in its quest to become more competitive, has found itself constrained to establish marketing channels at long distances and for extended periods. Even though refrigeration is one of the most commonly used preservation methods, it is insufficient to cover such needs. Therefore freezing becomes a solution in spite of its high cost compared to refrigeration and a decrease of the price of the thus preserved meat, which results in a paradox.

The primary requirement for a meat processor is to have and to be acquainted with equipment adapted for cost function and quality production. However, such a need cannot always be covered because the selection criteria employed are based on a company's capacity or facility to acquire such equipment. Nearly 80% of the water contained in fresh meat submitted to a  $-5^{\circ}$ C (23°F) freezing temperature is actually frozen; but it will rise to nearly 90% if it is frozen at  $-30^{\circ}$ C ( $-22^{\circ}$ F) (1).

The different types of equipment that have been utilized for the freezing of meat and processed products are presented in this chapter. Freezing equipment is classified according to its heat transference system (air, contact, immersion), its operation procedures (batch, continuous, or in-line) and its product handling presentation (before or after packaging).

#### II. FREEZING EQUIPMENT

There are three basic red meat freezing methods applied at the commercial level: freezing by air, freezing by immersion in a coolant medium, and freezing by contact. The type of equipment will depend on the method utilized. References for different types of equipment and their utilization for meat are shown in Table 1.

 Table 1
 Equipment Utilized in Freezing of Meat

Freezing method	Equipment	Application	Reference
By air	Chamber with plates	Meat carcass, cuts (with or without packaging)	(3, 4, 7, 11)
By air	Tunnel continuous	Meat carcass, cuts (with or without packaging)	(3, 9, 11)
Immersion	Liquid nitrogen	Meat in pieces, fish and chicken	(3, 6, 9, 11)
Cryogenic	Nitrogen atomizer	Meat, fish, and fruits	(8, 9, 11)
Contact	Horizontal plates	Meat cuts (packaged)	(3, 11)
Contact	Vertical plates	Meat cuts (packaged)	(3, 11)

# A. Freezing by Air

Such a freezing method is quite common; air is the refrigerating medium in it. Its particular compatibility with food even before packaging is one of its multiple advantages, and regardless of form or product dimension, it can be perfectly adapted to the processor requirements (3). This last issue turns into a very important matter in meat freezing because of the large bulks handled in certain cases (meat carcasses or large product boxes intended for ulterior transformation) (4). While freezing, cold air presents a low specific heat (1 kJ/kg°C), requiring just a small amount of energy to increase its temperature, which should be reflected in a low heat transference coefficient (25 to 35 W/m²°C) that is translated in a lack of heat transference in an air–product interphase. Air freezing devices are determined to be regularly voluminous due to these air properties (5).

Ventilation is an important factor in air freezing; since as the air speed increases, the amount of heat relinquished by the food by time unit increases as well, owing to an increase of the heat transfer coefficient. Therefore, in order to absorb the heat being relinquished by a product while freezing, great amounts of air must be handled. The energy cost required by a ventilator determines the practical limitations that indicate air maximum speed. It is interesting to analyze if this energy cost has an impact in the process, considering that this cost rises owing to an increase of air speed of the third power approximately. In practice, frozen air will normally be expelled at  $-30^{\circ}$ C if the fluid being frozen by the air (cryogenic fluid) reaches  $-40^{\circ}$ C. For the air to reach  $-40^{\circ}$ C it would be necessary to double the freezer energy consumption so that the freezer can lower its temperature (5).

Regarding red meat freezing by air, it can be carried out by utilizing the following procedures:

1. Cold storage rooms. There is no standard design equipment. These are chambers built specifically according to volume of the meat to be frozen (cuts or carcasses) and the presentation (with or without package). Basically, they are rooms designed to operate at low temperature levels. Airflow within chambers can be controlled in them, but not the air flowing over the product (6). It is worth mentioning that owing to air effects a product tends to dehydrate on its surface, which results in a mass loss and turns into an economical loss, especially if the meat has not been properly packaged.

Beef carcasses can be frozen in forced air freezers, which are usually refrigerated from 16 to 48 hours before freezing at temperatures from -10 to  $-30^{\circ}$ C and air velocities from 1 to  $3.5 \,\text{m/s}$ . At temperatures lower than bovine carcasses ( $-30^{\circ}$ C to  $-40^{\circ}$ C), pork can also be frozen with no previous refrigeration and so can lamb carcasses (without refrigeration) at  $-20^{\circ}$ C at an air speed of from 0.5 to 1 m/s for 12 to 18 hours. Regarding meat cut freezing, for either processing or separate selling, the most common way is by using individual packaging or grouped by muscle type placed in 15 cm width boxes. Temperatures of  $-30^{\circ}$ C and air velocities of  $12 \,\text{m/s}$  are the conditions utilized (5).

2. Tunnel types. These are chambers equipped with vaporizers and ventilators, where cold air circulates through the products located on trays or in boxes from 20 to 30 kg. Such boxes and trays are prepared on stationary shelves or on cars that travel through the tunnel, either isolated or in series, separated enough so the air can freely circulate. As red meat freezes, it is suspended over transporters or rails (6, 7). Cold air flows perpendicularly to a product movement, providing air in the tunnel in continuous sections. This represents the advantage of having only one way in and out for its unloading. Having a perpendicular cold air direction allows a heat exchange reduction between the air within and outside the tunnel. Another advantage is that thermal conditions to keep humidity level high in each section can be controlled, avoiding evaporation and preventing dehydration on the product surface, which leads to freezer burn. Thermal transportation in this type of freezing depends on dimensions and product form. Tunnel freezing equipment is very flexible and adaptable to products of all dimensions and forms, packaged or not (4, 8).

# B. Freezing by Immersion

Direct contact between the food or the container that holds the product and the coolant is established in this type of freezer. Methods of freezing can be by submerging the food in a cold liquid or spraying the liquid on it, using brines and syrups as well as calcium chloride, ethylene glycol, propylene glycol, or alcohol among others (6). This method has been in practice since the beginning of the freezing process. In freezing by direct immersion, coolant selection becomes limited, especially when the food is not covered by package. Thus the coolant must be nontoxic, of a certain purity, and free of coloring, and flavors, strange odors. Also, when it comes to packaged foods, it is important that the coolant does not contain products or substances that could corrode the container (9). Nowadays, this freezing method is employed in products of irregular form such as meat, fish, and chicken (4).

# C. Cryogenic Freezers

In cryogenic freezing, direct contact between the coolant and the food takes place. This has proven to be a good conservation method. In the equipment the coolant is not provided by a static cooling system; it is a consequence of the change of phase from a heat-transmitting agent. Such an agent can be a sublimatory solid (solid carbon dioxide) or a cryogenic liquid (a liquefied gas with an extremely low boiling point) such as liquid nitrogen at -196°C or carbon dioxide at -78°C (5, 10).

Liquid carbon dioxide under pressure is employed to reinforce conventional freezing lines by cold air or to provide a total refrigerating medium in a freezing room. A mixture of cold carbon dioxide and carbonic snow (solid) is produced when this liquid atomizes, which can establish contact with the food to freeze. Since liquid nitrogen's boiling point is  $-196^{\circ}$ C (at atmospheric pressure), freezing velocities are extremely fast. This provides a

quality hardly reached by any other freezing method known. High heat transference coefficients between a solid and a coolant fluid can be obtained by using liquid nitrogen. Irregularly formed products can be frozen as easy as rectangular blocks, as they can equally be frozen individually (individually quick frozen, IQF). A frozen food production by this method offers an advantage, since it allows the consumer to acquire individual portions from a package, storing the rest until needed (3, 11).

Nitrogen is the most important cryogenic liquid used in freezing by immersion. It is employed for meat cuts, fish fillets, seafood, fruits, etc. The employment of this cryogenic liquid is common in freezing equipment through tunnels. A reference to several types of equipment used appears in Table 1.

The most commonly used equipment for this type of freezing is the one in which a product enters from one side of the tunnel and nitrogen is sprayed from the other; when it vaporizes, cold nitrogen gas runs through the tunnel until reaching the product. The gas lowers product temperature before the sprayed nitrogen gets to it. Once in contact with the spray, its superficial temperature goes down to a value close to nitrogen temperature (-195°C). The product passes to an equilibrium zone where the cold surface temperature and the hottest core temperature are equalized in order to reach uniform temperatures. Finally, water spraying is applied to induce the cold stored within the product to form a thin frost layer, which will protect it from storage dehydration. Immersion freezing equipment is more commonly used to freeze packaged white meat (chicken and turkey) than for red meats.

Generally, compared to any other method, liquid nitrogen freezing causes fewer losses by freezing dehydration and thawing drainage. This can represent up to 5% of the weight in some foods. If analyzed, this freezing process, though expensive, can prove profitable considering the quality and volume obtained, for instance, in large-scale beef patty production. An advantage of this type of equipment is that its installation expenses are lower than others, approximately 30% less than any freezing system of the same capacity. Weight loss by product dehydration is reduced (8).

# D. Freezing by Contact

Freezing by contact includes methods in which food or food containers are in contact with a surface cooled by a refrigerant. When this method is utilized, the food or container is not in direct contact with the coolant (5, 12). Generally, it requires a flat surface or an almost flat surface to be in contact with the refrigerated plates for solids or packaged products. Thus contact between these plates and one or two surfaces of the food or food container can be established (9). A good operation of this type of equipment is achieved if the heat transmission coefficient between food and coolant is high and uniform. This is possible if there is deep contact from the food to the package material, that is, if the vessel material is completely full of food. Aluminum compounds employed for plate manufacture have improved freezer plates capacity significantly (3).

Contact equipment freezing time periods are shorter than for air freezing systems; for instance, cardboard boxes with 27 kg of meat are frozen in from 14 to 16 hours instead of 20 to 24 hours in a freezing tunnel (13).

Freezing by contact can be made with equipment that operates with plates provided in a horizontal or vertical way. In both cases such devices assure a good thermal transmission in a short freezing period, if the product itself is a good heat conductor. The advantage of this transfer is reduced when product thickness increases; this procedure is more frequently applied to a thickness that does not surpass 5 or 6 cm, limiting its use to

meat cuts packaged over trays (4, 6). Although it is possible to place solid foods over an ice block or dry ice, it is rarely done at a commercial level. What is actually done is placing the food over plates, trays, transporting bands, or even more cold walls frozen by a circulating coolant, so that the food is kept in contact with a cold wall and indirectly in contact with the coolant. A more detailed description of equipment provided with horizontal and vertical plates is presented next (4, 10, 14).

- 1. Horizontal plates. In this case, a product is placed on trays or metallic tables, which can be accomplished manually or automatically. Automatic operation is carried out by doing the following: The battery from the plates can be moved upward or downward in an elevating system; at a load carrier level, plates are separated and the packages accumulated on the conveyor are pushed among the plates, unloading simultaneously frozen packages on the opposite side. The operation is repeated until all the frozen packages have been replaced; the two plates are adjusted again afterwards, the set goes back up, and a new cycle begins.
- 2. Vertical plates. They are mainly used for products in blocks from 10 to 15 g, such as entire fish or meat cuts. This equipment contains a set of cooled plates, which at intervals form the compartments where the product is placed. The frozen blocks are unloaded sideways, upwards, or downwards.

Certain freezing processes by indirect contact must not be mistaken with direct immersion. In order to distinguish one from the other, a direct contact from the food or its vessel with its coolant must be apparent. It will be a direct immersion freezing if a slice of meat or a slice of meat covered by a film is submerged in a brine bath or if it is sprayed with it. If the same meat is placed in a metallic bucket and immersed, the food and its package are being frozen by indirect contact.

### III. EQUIPMENT SELECTION

A wide range of equipment to freeze foods is available in the market. Thus there is a long list of characteristics that must be fulfilled in order to select a freezing method and freezing equipment. Until now a final selection has depended basically on the cost-benefit analysis that it represents. However, there are many factors affecting the adoption of a correct cooling installation, and it is difficult to make the right decision. Among the main factors are (6, 7, 15):

- 1. An appropriate size or capacity for the production
- 2. Space requirements
- 3. Cost and benefits for equipment installation and maintenance
- 4. Quantity of products and presentations that can be frozen
- 5. Cooling power (Q) required
- 6. Operation temperature from the cooling fluid  $(T\infty)$
- 7. Heat transmission coefficient (h) achieved for freezing operation (Table 2)
- 8. Energy recovered as well as freezing as part of the process in line
- 9. Feasibility and method comparison regarding evaporation losses

Furthermore, some inherent characteristics must be considered:

- 1. Process: Damage to the product, hygiene, and safety (6)
- 2. Product: Composition (humidity, w), thermal conductivity ( $\lambda$ ), specific heat (C), specific weight ( $\rho$ ) and geometry (5, 12).

**Table 2** Heat Transfer Coefficient of the Surface Layer According to Freezing Method

Freezing method	Heat transfer coefficient W/(m <sup>2</sup> °K)
By airflow	
static	6–9
air at 3 m/s	18
air at 5 m/s	25–30
By immersion	
in freon	500
in liquid nitrogen	1500
By direct contact	
plates	100

Source: Adapted from Refs. 8, 15.

The equipment selection definitely depends also on the size of the company in a sense that a smaller company will give more credit to a direct cost-benefit analysis along with characteristics of the freezing product to freeze. On the other hand, in a medium-size company, besides these factors, the points listed before must be analyzed and prioritized according to the expected benefits. In big companies, these factors are completely taken into account and include some more of their own as to effort, personnel, machinery, and storage time saving policies. However, all these factors become obsolete if an inventory of the freezers is not done. Good handling practice, which is using the right procedures or systems for in and out product control (just on time, in and out frozen products, etc.). It is common to hear that "freezing is useless because it decreases products quality with its usage" when problems arise, but it is not completely accurate. Therefore it is important to train the commercial user on the equipment, operating conditions, and products to freeze.

#### IV. CONCLUSIONS

The different types of equipment utilized in meat freezing were described in this chapter. Regarding carcass freezing and boxed meat, the use of air remains the most common method at the commercial level, especially because it represents a lower cost concerning the volumes handled. It is also worth emphasizing that the utilization of contact freezing and the use of cryogenic agents offer greater advantages based on the ultimate quality of the product being frozen. Therefore its usage is still limited to cut meats, where the operation cost is covered by the sale of the product. In conclusion, a meat freezing equipment selection must be very carefully carried on and one must consider not only cost—benefit analysis but also priority factors according to size and company policies.

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# 12

# Frozen Meat: Quality and Shelf Life

M. L. Pérez-Chabela

Universidad Autónoma Metropolitana, Mexico City, Mexico

J. Mateo-Oyagüe

Universidad de León, León, Spain

### I. INTRODUCTION

The main objective of freezing is to prolong the shelf life of foods. On this basis, freezing is considered an excellent means of maintaining acceptable quality in red meats for months and even years.

Currently, consumers prefer chilled to frozen red meat, probably because of a desire on the part of the purchaser to assess several meat quality attributes, such as the ratio of bone to muscle, the presence of fat, and the like (1). Such an assessment is difficult when meat is frozen. Despite this, freezing and frozen storage of red meat is gradually increasing. Frozen storage is regarded as a useful technological aid because it makes possible the supply of large amounts of meat for catering and supermarkets, extends the storage life of meat when supply exceeds demand, and represents a flexible tool for sellers.

The quality of red meat brings together sensory (i.e., texture, flavor, and color), microbial, nutritional, and functional characteristics. In most cases, freezing does not modify quality in any considerable way. However, frozen meat may present defects in quality and naturally can suffer deterioration in quality during storage. Color, flavor, and texture defects, together with moisture losses, are the principal factors to which due regard must be paid.

Color and flavor can become altered during storage, with lipid and myoglobin oxidation being the main adverse changes affecting the eating quality of frozen red meats.

With respect to texture, there are two circumstances having detrimental effects on the quality of frozen meat, both related to a significant decrease of temperature in prerigor meats. These are cold shortening and thaw rigor, which lead to toughness. In order to avoid these processes the advice has generally been to freeze meat after rigor instead of before it. Nevertheless, prerigor freezing is also possible, provided that preventive measures are brought into play, such as electrical stimulation and tempering during thawing.

In addition, frozen meat production implies some profit losses related to the reductions in weight resulting from freezing. Generally, in comparison with rapid freezing, slow freezing produces greater damage to the muscle structure, resulting in a decrease in

myofibrillar protein solubility and water holding capacity (WHC) and an increase in weight losses on defrosting or thawing (2).

The principal factors affecting frozen meat quality include the intrinsic quality of the meat, chilling and freezing conditions, and protection and storage conditions throughout the cold chain (3–5) (see Table 1). These factors are not independent of one other. The effects on the eating quality of red meats of the aging time of meat before freezing, the freezing rate, and the cooking method used, and also on thawing and cooking losses, showed a large number of significant interactions (6).

In relation to the effect of storage temperature on the quality of frozen foods, the glass transition temperature under conditions of maximal freeze concentration (with a slow-frozen sample), Tg', is a potential means of assessing the stability of foods that deteriorate as a result of diffusion-limited events. In the case of beef, apparent Tg' values were approximately  $-12^{\circ}$ C (7). This temperature seems also to be the lower limit for microbial growth in meat.

In this article, the topics reviewed are the structure, moisture losses, microbiology, organoleptic properties, nutritional value, and shelf life of frozen red meat.

# II. STRUCTURAL DAMAGE IN FROZEN MEAT (EFFECTS OF FREEZING RATE AND FREEZER TEMPERATURE)

Muscle cells are flexible and elongated, share their alignment, have minimal air spaces, and are separated by a matrix rich in glycoproteins. Although muscle cells are difficult to rupture by freezing and thawing, freezing and frozen storage can produce marked effects on the structural properties of red meat tissues, with cell–cell separation being the most evident structural change.

The freezing point in lean red meats is approximately  $-1.5^{\circ}$ C, depending on meat composition. After this point, as temperature decreases, a higher proportion of water turns into ice crystals. At temperatures below  $-7^{\circ}$ C more than 80% of the water is frozen. Water outside the muscle fibers freezes first; thus intracellular water tends to be drawn out of the fibers by osmotic effects, increasing the intracellular concentration of solutes. The consequences of this cryoconcentration (i.e., changes in osmotic pressure, pH, ionic force, viscosity, Aw) trigger the denaturing of and damage to muscle (i.e., myofibrillar and sarcoplasmic) protein, which progress with longer time and lower temperature of frozen storage (8). These changes in the functional properties of proteins may be responsible for

**Table 1** Main Considerations Influencing Frozen Red Meat Quality

**Prefreezing**: species, genetic, livestock, feeding—lipids, vitamin E, transport, slaughtering. Postmortem processes—electric stimulation, chilling, hot deboning, freezing pre- or postrigor, aging. Quality of raw materials including microbial status, type of muscle, processing of meat—cutting, cooking, packaging

Freezing: freezing rate, freezing technology

**Frozen storage:** temperature, time, temperature fluctuations **Thawing:** rate, technology, tempering or cooking of frozen meat

Culinary elaboration: technology, temperature, time

Source: Adapted from Ref. 4.

changes in texture, color, and flavor. Finally, as solutes reach the saturation point, two outcomes are possible: the formation of eutectics or of a supersaturated metastable rubbery phase (glass transition). According to Fenema (9), the formation of eutectics during the freezing of meat is uncommon.

Freezing rate has an important influence on crystal size, location (intraor extracellular), and morphology. Slow freezing rates (i.e., a rate of advance of the frozen front below the range 0.1–0.2 centimetres per hour) in matured meats produce large and, according to Grujic et al. (10), for the most part extracellular crystals, which damage muscle proteins and cell membranes, causing considerable distortions in tissue microstructure. Thus the WHC of the meat decreases, and the exudate produced during thawing increases. At intermediate freezing rates (from 0.2 to 1.0 cm/h) medium-sized ice crystals are formed both inside and outside cells. In this case cellular damage and drip losses are also high. By contrast, fast freezing rates (1.0 to 10.0 cm/h) bring about the formation of numerous small crystals, mainly intracellular, uniformly distributed. In this case, there is a minimum of physical harm to cells and of chemical changes to proteins (11). However ultrafast freezing (in excess of 10.0 cm/h) may cause freeze-cracking in joints of frozen meat owing to sudden changes of volume. The full effects of fast and slow freezing on the eating quality of frozen meat are still unclear. In general, it is considered that freezing rate has only limited effects (4).

As storage times get longer, ice crystals undergo gradual growth (12). Temperature fluctuation is considered a major stimulator of crystal growth through inducing recrystallization. Crystal growth is associated with a decrease in the WHC of meat protein, an increase in tissue damage, and a subsequent increase in drip losses from thawing meat.

In order to reduce structural damage during freezing, a number of cryoprotectants have been used in meat, including aspartate, cystine,  $\beta$ -alanine, sugars, dicarboxylic acids, phosphates, and starch hydrolysis products. Unfortunately, the use of cryoprotectants has sometimes been associated with a sweet taste or lipid oxidation defects in meat. Nonetheless, these substances are used in cut, minced, and restructured meat, and some of them have even been incorporated into meat by injection in live animals before slaughtering (13). Hence a topic of great research interest is the application of antifreeze glycoproteins found naturally in cold-water fish or similar substances, incorporated in meat by brining or injection. These substances have the ability to make crystals smaller in size, to inhibit recrystallization and so to reduce drip loss.

#### III. MOISTURE LOSSES

During freezing, storage, and thawing, meat loses water by evaporation, sublimation, and exudation, respectively; moisture is also lost during cooking. Moisture losses are of great monetary importance. Although moisture losses make meat less attractive, they do not significantly influence its eating quality after cooking, except in the case of very large losses, which could affect juiciness and tenderness (4).

Evaporation losses depend on freezing conditions [relative humidity (r.h.) and temperature] and meat characteristics (the size and area-to-volume ratio of pieces, the fat covering, and the presence of skin or packaging). Moisture losses by evaporation during freezing of nonpacked carcasses or joints normally amount to between 0.5 and 1.2% of the total weight (4).

Sublimation during storage is another important cause of moisture loss. Greater weight losses occur when there is a higher air speed, more surface of lean exposed, longer storage, or a higher temperature. Monthly losses of 0.15% to 0.7% were found in traditional stockinette-wrapped meat stored at  $-30^{\circ}$ C and  $-10^{\circ}$ C, respectively (14).

The exudate formed during thawing (purge or drip loss) generally accounts for between 1% and 5% weight of meat pieces (4). Drip loss has a negative financial impact on processors, makes the meat visually unattractive, and also involves loss of soluble nutrients. Amounts of drip loss from frozen meat are determined by two main factors: the volume of the exudate generated during defrosting and its migration speed.

The former depends on the freezing and thawing conditions and features intrinsic to the meat itself (15–17), the water-binding capacity of proteins being involved in both. The latter depends on the size and shape of the pieces of meat, the myofibrillar orientation and geometry, the presence of large blood vessels, and the intensity of physical cellular or macroscopic damage (freeze cracks) produced during freezing, that is, with fast freeze rates.

As previously mentioned, drip loss has been associated with cellular damage caused by large crystals and extracellular crystal presence, these crystals being formed at slow and intermediate freezing rates. Furthermore, the water from the extracellular crystals formed seems not to be reabsorbed on thawing, thus producing drip loss. Hence intermediate and slow freezing rates (those in which the time taken for the temperature to fall from  $-1^{\circ}$ C to  $-7^{\circ}$ C exceeds 10 minutes) show greater drip loss than faster rates (15). Within this range, these authors found a peak in drip losses in defrosting at a rate of 15–20 min. In the light of the effect of freeze rates on drip loss, several authors recommend freezing rates in the range 2.0 to  $5.0 \,\mathrm{cm/h}$  (10).

When industrial freezing methods based on thermal gradient are used, large volume carcasses and joints undergo different freezing rates depending on depth, so that the thermal center shows larger and more numerous extracellular ice crystals, which lead to a poorer quality product (18, 19). New methods like high-pressure-assisted freezing (for example at a pressure of 200 MPa) would be necessary to overcome these difficulties (12). Pressure brings the freezing point of water down to a minimum of  $-22^{\circ}$ C at 207.5 MPa (20). Thus meat would be cooled to  $-20^{\circ}$ C under pressure, so included water remains in its liquid state, and then pressure would be released, so that an instantaneous and homogeneous microcrystallization would occur.

In addition, high and fluctuating temperatures and long storage favor large crystal formation and hence increased drip loss. Valin et al. (1971), cited by Genot (4), found drip losses in vacuum-packed pork, beef, and veal of 1.2%, 4.2%, and 2.2%, respectively, after four months' storage. When storage was 16 months, the losses were 4.7% for pork and more than 8% for beef and veal. In other work, it has been reported that drip losses from fast-frozen and stored meat increase with storage time, reaching the same level as the drip losses observed from meat frozen at a slow rate but not stored for any length of time (21).

Packaging also shows an influence on drip loss, vacuum- and oxygen-impermeable packaging being the most effective. For instance, the volume of exudate from cut pork kept frozen for 13 weeks at  $-17^{\circ}$ C was between 2.5% and 5.0%, in accordance with the packaging characteristics (22).

Moreover, thawing time seems to be correlated with extracellular water reabsorption, which is a slow process. This might be the reason that faster thawing rates (defined as involving a time of under 50 min. for the temperature to rise from  $-5^{\circ}$ C to  $-1^{\circ}$ C) produced more drip loss in frozen beef (23). In contrast, other authors found that fast thawing rates resulted in smaller drip losses (16, 21). The most recent suggestion has been

that slow thawing causes more structural damage through recrystallization (24) and more protein denaturation. These authors recognized that fast freeze rates were more suited to fast thaw rates and vice versa.

Several intrinsic factors have been noted as affecting drip loss: species, type of muscle, pH [which has an inverse correlation with drip loss (17)], prerigor and postrigor freezing (the latter presenting more exudate in the case that thaw rigor is avoided), or aging time (matured meat gives less drip loss).

To sum up, under industrial freezing conditions, storage time, temperature fluctuations, and the intrinsic characteristics of the meat, rather than the freezing rate, seem to have the biggest effects on drip loss. Finally, cooking loss from frozen meat depends principally on the processing of meat before freezing, especially rigor onset temperature (25), and on the cooking method, particularly the cooking temperature. Although cooking loss is accepted as being higher when freezing rates are slow (see the review of Genot), the effect of freezing rate on cooking loss seems to be slight (26).

## IV. MICROBIAL EFFECTS (HYGIENE)

Low freezer temperatures (i.e., below  $-18^{\circ}$ C) bring about a practically total inhibition of the cellular metabolism in animal tissues and substantially decrease the rate of almost all chemical reactions. Microbial development in meat virtually comes to a stop when a temperature of  $-12^{\circ}$ C is reached (17), this being the lowest point reported as allowing growth of molds on meat. In this way, mold spoilage may be the most substantial problem affecting frozen carcasses that have been subjected to temperature abuse.

In general, a large proportion of the microbiota of a foodstuff will be killed or sublethally damaged by freezing (owing to thermal shock, ice formation, dehydration, or solute concentration). In fact, a decrease in aerobic spoilage organisms has commonly been observed in frozen meat (27). Freezing process and animal species involved influence the microbial counts. Faster freezing rates have been demonstrated to have a less injurious effect than slower ones, and in addition, the longer the storage time, the more serious the damage.

Among the various organisms, gram-positive bacteria are generally more resistant to freezing than gram-negative, cocci more than bacilli, and yeast and molds more than bacteria. Bacterial and fungal spores and toxins of *Clostridium botulinum* and *Staphylococcus aureus* are highly resistant to freezing (28). In any case, pathogenic microorganisms are commonly isolated from thawed frozen meats. Furthermore, freezing has a sanitizing effect on frozen red meat. Freezing can be used to destroy larvae of *Taenia* spp. and *Trichinella spiralis*. These organisms are killed after 1 to 3 weeks at  $-18^{\circ}$ C or after an ultrarapid freezing at  $-29^{\circ}$ C (29).

Nonetheless, defrosted meat seems to spoil faster than chilled meat because of the damage to tissues and because the exudate formed enhances microbial growth (4). Thus the thawing process represents a fundamental phase with respect to the microbiological quality of thawed meat. In this way, increases in bacterial numbers become unacceptable in the air thawing of beef and pork carcasses or quarters if temperatures in excess of  $1^{\circ}$ C are maintained (5). Also, the time for which meat surfaces are at temperatures in excess of  $1^{\circ}$ C must be kept short if pathogen development is to be prevented. In this respect, tempering meat pieces during thawing at temperatures between  $-5^{\circ}$ C and  $-2^{\circ}$ C, and processing the meat (undertaking operations such as cutting, mincing, and the like) at these temperatures may be really advisable. Apart from thawing, microbial counts in

frozen meats depend on the initial level of microbial contamination and contamination during freezing and storage.

The microbiological status of good-quality meat [a satisfactory standard has aerobic plate counts at 25°C from the surface of frozen prime joints of beef under 10<sup>5</sup> colony-forming units per square centimeter (cfu cm<sup>2</sup>) and coliform counts under 10<sup>3</sup> (30)], and of frozen minced beef under 10<sup>5</sup> and under 10<sup>2</sup>, respectively (27), is largely due to hygiene in dressing and butchery of the carcasses, short time and low temperature of chilled storage, high freezing rate, proper packaging, and so forth.

# V. ORGANOLEPTIC PROPERTIES OF FROZEN RED MEAT

# A. Texture (Cold Shortening and Thaw Rigor)

In frozen red meat, changes in the main attributes of meat texture, namely tenderness and juiciness, depend principally on changes in myofibrillar and stroma proteins (rigor shortening, denaturation, cross-linking, linking between proteins and free fatty acids or lipid autooxidation products and similar). These changes affect the functional properties of proteins (WHC, solubility, and enzyme activity) throughout the freezing and thawing processes. The severest effects appear at low freezing rate and long-time high-temperature storage (31, 32). However, the mechanisms of protein denaturation and other changes in frozen muscles are still not fully understood, and their real effects on meat eating quality have not been totally established (33).

The freezing process for postmortem meat in industrial practice seems to have only a slight effect on tenderness and juiciness. These attributes are more dependent upon the intrinsic factors of meat, specifically maturation before freezing, which improves tenderness. On this point, it has been found (34) that beef frozen after 14 days of aging was the tenderest.

In any case, there have been several instances in which meat frozen and then thawed was more tender than chilled meat (35, 34). This may represent an advantage for beef and lamb eating quality. The tenderizing effect of freezing might be explained by tissue damage due to ice formation (36) or an improved aging process in red meats resulting from a decrease in calpastatin activity (37).

On the other hand, prerigor freezing could effectively produce a detrimental effect on meat tenderness, if cold shortening and/or thaw rigor took place. Thaw rigor may account for 40% of myofibrillar shortening (17), which induces toughness. These effects are not frequent in entire carcasses of pork and beef, especially if they are covered by a layer of fat and the freezing rate is not too fast (17), for rigor occurring before the decrease in temperature becomes critical. However, in lamb carcasses and hot-deboned joints both processes can occur. In these cases, lower freezing rates [i.e., attaining  $-1^{\circ}$ C within 5 hours postmortem (18)] are responsible for cold shortening and higher ones for thaw rigor, which is the case of meat frozen before the onset of rigor mortis.

Nonetheless, cold shortening and thaw rigor can be prevented. Postmortem electrical stimulation, which accelerates the glycolytic process and hence the onset of rigor mortis, has been used to prevent, and can effectively prevent, cold shortening and also thaw rigor (17, 36). For instance, electrical stimulation of lamb carcasses prevented cold shortening at freezing rates such as those needed to attain  $-4^{\circ}$ C within 12 hours postmortem [Devine et al. 1996, cited by Genot (4)].

Besides, if meat is frozen before rigor onset, tempering the meat at temperatures between  $-2^{\circ}$ C and  $-5^{\circ}$ C for approximately three days during thawing can prevent thaw

rigor. At these temperatures, the ATPase activity is enough to trigger rigor in the frozen state, thus preventing shortening. ATPase activity has been observed even at  $-12^{\circ}$ C (18). Another way of preventing thaw rigor consists of adding salt, ca. 1.5–1.8%, to meat pieces (38). Salt inhibits glycolysis and rigor mortis onset, thus the levels of ATP, pH, ionic force, and WHC of meat remain high during the freezing and thawing processes.

To conclude, provided that the measures to prevent cold shortening and thaw rigor are brought into play, prerigor frozen meat can be better than postrigor, that is, prerigor frozen meat has been reported to preserve to a great extent the functional properties of proteins and produce less drip loss during thawing and cooking (39, 40).

# B. Flavor and Color (Lipid Oxidation and Discoloration)

Raw meat has a weak or "flat" flavor, but it contains flavor precursors from which more than a thousand volatile substances are generated during cooking by a complex sequence of chemical reactions. It appears that a freezing—thawing process applied to beef loin (34), or to red meats in general, has no significant effect on flavor. However, during frozen storage, the flavor of meat can be somewhat altered. Firstly, flavor seems to decrease and lose balance but does not become unpleasant. In this regard, Lawrie (17) pointed out that there is a gradual loss of the most volatile compounds even at temperatures below freezing. Also, flavor and flavor precursors that are soluble substances (reducing sugars, amino acids, peptides and nucleotides such as IMP and GMP) may be lost or degraded during freezing and above all during thawing. Equally, freezing-related protein changes can account for differences in their ability to bind taste and volatile compounds, thus modifying flavor perception.

Finally, after longer storage times unacceptable "off" flavors may be detected, normally rancid flavors, which are produced by numerous volatile compounds coming from lipid oxidation. This process can be avoided in full only if oxygen is completely eliminated and storage temperature is extremely low, i.e., under  $-60^{\circ}$ C (41). Furthermore, flavor may be altered by interactions between several oxidation products and different flavor compounds, or hydrophilic peptides (42). Apart from flavor alteration, lipid oxidation may affect texture and nutritional properties of frozen meat. Lipid oxidation is definitely one of the principal limiting factors for frozen meat shelf life.

Theoretically, factors favoring or inducing meat lipid oxidation are numerous—traces of transition metals, sodium chloride, light, heat, protons, active oxygen species, damage to muscle structures, and enzymes. These enzymes (lipases, lipooxigenases, and so forth) may remain active at subzero temperatures (9,43). On the contrary, muscle possesses endogenous antioxidant capacity (involving  $\alpha$ -tocopherol, carotenoids, ubiquinone, glutathione, carnosine, anserine, ascorbic acid, and the enzymes with antioxidant activities, such as glutathione peroxydase, superoxide dismutase, catalase). Storage of meat may result in a decrease in the activity of the antioxidant enzymes (44).

During freezer storage, when the balance of the factors favoring oxidation and of the antioxidant capacity of muscle foods tilts towards lipid oxidation, the latter is no longer controlled. In these circumstances lipid oxidation begins, first in the highly unsaturated phospholipid fraction in cellular biomembranes, thanks to its amphiphilic nature and proximity to the aqueous medium (45). In addition, triglycerides and cholesterol can be oxidized.

On the other hand, visual attributes such as red bright color, fat color, fat content (including perceived marbling and external fat cover), and general appearance serve as the

first or only consumer indicators of the quality of chilled, and where possible frozen, red meat at the time of purchase.

Browning of meat during frozen storage and thawing is a consequence of myoglobin oxidation, which turns the pigment to metamyoglobin. If the percentage of metamyoglobin on the meat surface reaches a certain point (ca. 40% of total myoglobin), purchasers will reject meat. Accumulation of metamyoglobin depends on the relative rates of oxymyoglobin autooxidation and enzymatic or nonenzymatic reduction of metamyoglobin to oxymyoglobin. The rate of autooxidation of myoglobin is strongly dependent on temperature and exposure to light. Unlike chilling, where thermal autooxidation is predominant, under freezer storage conditions, light-induced processes become increasingly important (46, 47). Oxygen concentration is another important factor for color stability. According to Lanari et al. (48), vacuum packaging and packaging material with incorporated light adsorber increase frozen red meat shelf life. Furthermore a blooming time (some 6 to 48 hours) in oxygen before freezing is advisable Color instability together with lipid oxidation definitely constitute the principal limitations on frozen meat shelf life (Table 2).

Brown discoloration (metamyoglobin formation) and lipid oxidation in frozen red meats seem to be related (49, 50). Thus Lanari et al. (48) and Guidera et al. (51) claimed that frozen meat color stability and susceptibility to rancidity indicated the oxidation status of meat. Both processes demonstrate similarities in their progression in meat and meat products. Experimental results support the view that lipid oxidation contributes to oxymyoglobin oxidation and meat discoloration (52). However, in frozen meats, color losses generally occurred much earlier than lipid oxidation (47, 49); these authors state that pigment oxidation might be an initiator of lipid oxidation. In fact, the exact mechanism for a possible coupling between pigment oxidation and lipid oxidation is not well understood (5).

The susceptibility of specific frozen red meats to lipid oxidation and discoloration is influenced by intrinsic and extrinsic factors, which are shown in Table 3 (adapted from Ref. 4).

Frozen meat color at the surface may also be affected by excessive dehydration during storage, appearing as grayish-brown leathery spots: freezer burn, which leaves the meat dry and stringy. Moreover, dehydrated surface conditions enhance protein denaturation and lipid and color oxidation. Freezer burn is not necessarily correlated with moisture loss in a simple manner (53). It tends to happen when meat surfaces are initially wet and when freezing is very fast, when the lean meat surface is not protected, air speed in storage chambers is fast, and there are temperature fluctuations. In order to

**Table 2** Times of Storage (Months) at Which Lipid Rancidity or Browning Are Manifested in Lean Meat

	Storage temperature			
	-8 °C	−15 °C	−22 °C	−30 °C
Beef	3	6	12	
Pork without skin and fat cover		3	6	12

Source: Adapted from Ref. 17.

**Table 3** Intrinsic and Extrinsic Factors Influencing Lipid Oxidation and Discoloration in Frozen Meats

Factor	Several significant effects	Main causes
Species differences <sup>a</sup>	Intrinsic Pork is more sensitive than beef	Pork has more PUFA <sup>b</sup> than beef
Animal to animal variation: genetic, season, physiology		Not fully understood
Feeding	The more the PUFA—or linoleic acid contents—in the diet, the more unstability, especially in monogastrics. Higher content of antioxidant or prooxidant compounds in the diet represent an increase or decrease in stability, especially vitamin E.c	Meat composition: vitamins E and C, beta-carotenes, carotenoids, prooxidants such as Cu and Fe, oxidative status of lipids in the feed
Cuts of meat and muscle type	Red muscles are unstable than white ones.  Extrinsic	Oxidative pattern and composition differences, i.e., pH, % PUFA, antioxidant dipeptides (43)
Transport and slaughtering conditions	Stressed lamb carcasses with high pH were unstable to rancidity. (Al-Dulamy and Aswad, 1987, cited by Haard, 1997).	Variations in pH
Chilling conditions, time and aging	Increasing the time in chilling reduces frozen storage life. Hot-deboned frozen meat is more stable (Andersen y col. 1991).	
Processing of meat		
Cutting and muscle disintegration, d structure damage	It favors the deterioration reactions contributing to reduced shelf life.	Enzyme/substrate contact is facilitated
Salting	Salt contributes to unstability in frozen red meats, especially with light exposure (Akamittah y col., 1990).	Amplifies Fe reactivity in autooxidation, modifies enzymatic activity (44), may be a source of traces of prooxidant compounds (47)
Pre-cooking <sup>e</sup>	On the one hand, precooking increases the propensity of meat to undergo oxidation during freezer storage. On the other hand, cooking may help to increase shelf life (George, 2000).	Cooking produces a structural change and accounts for liberation of prooxidant compounds

 Table 3
 Continued

Factor	Several significant effects	Main causes
Antiooxidants addition in meat (mainly minced or restructured)	Tri-polyphosphates (TPF), fenolics—i.e., BHT, TBHQ, BHA, propyl gallate <i>d</i> -tocopherol (Faraji y col., 1991), spices—i.e., rosemary, marjoram, cumin, ginger (El-Alim y col., 1999), carnosine at 0.5–1.5% (w/w) (Dekker y Crum, 1991).	Antioxidant activity
Packing/wrapping	Proper packaging <sup>e</sup> increased stability. This stability may represent the same as a decrease in 10°C temperature of storage from -10 to -20°C (Jull, 1984, cited by Genot, 2000).	Oxygen and light barriers
The freezing process	•	
Freezing rate	Freezing rate has not shown a significant effect.	
Storage temperature	The lower the temperature, the higher the stability.	Oxidation reactions are temperature dependent
Temperature fluctuations <sup>f</sup>	Oxidation is favored.	
Thawing conditions	Lipid oxidation may suppose a problem in long thawing processes at subzero temperatures (17) <sup>g</sup> .	

<sup>&</sup>lt;sup>a</sup> Lipid oxidation has been an important problem in pork as well as comminuted lamb and beef ((8), Rhee, 1996).

<sup>&</sup>lt;sup>b</sup> Unstability of frozen meat to lipid oxidation depends mainly on polyunsaturated fat acids (PUFA) content.

<sup>&</sup>lt;sup>c</sup> Supplemented vitamin E in diet—usually α-tocopherol—at supranutritional level, i.e., >100 mg/kg feed during 50 days before slaughtering, accounts for a significant increase of vitamin E content in muscle, reaching levels of 3.5–7 mg/g (Wulf y col.). At this amount, vitamin E appears to delay oxidation processes during frozen storage (Monahan y col., 1994; Liu y col. 1995b; Lynch, 1999). This protective effect against oxidation of vitamin E might be reinforced with added antioxidants and vacuum packaging in order to eliminate practically the risk of oxidation of meat. Furthermore, additional amounts of vitamin E in diet were responsible for a decrease in drip loss in pork (Asghar y col., 1991; Cheah y col., 1995).

<sup>&</sup>lt;sup>d</sup> Mechanically deboned meat is especially unstable.

<sup>&</sup>lt;sup>e</sup> The rule of thumb to achieve a long shelf life in cooked-frozen meat consists of oxygen- and light-barrier packaging materials, i.e., aluminum foil, polyvinidene chloride, etc., and storage below  $-18^{\circ}$ C.

<sup>f</sup> Temperature fluctuations cause the rate of several changes to increase or decrease, especially in the vicinity of

<sup>&</sup>lt;sup>1</sup> Temperature fluctuations cause the rate of several changes to increase or decrease, especially in the vicinity of the freezing point, the glass transition point, and other phase-transition temperatures.

<sup>&</sup>lt;sup>g</sup> Thawing must be fast enough to delay oxidation reactions but slow enough to permit water reabsorption.

prevent freezer burn the recommendation is to prevent air from reaching the surface of the meat, mainly by using vapor-proof packaging materials.

Other defects in color and/or aspect of frozen meats are yellow coloration of fat through lipolysis, deformations and freeze cracks arising from sudden changes of volume during freezing, and mold development caused by temperature abuse.

Finally, meat may show different colors according to the size of the ice crystals. When the freezing rate at the surface of the meat is high, crystals are smaller and scatter more light than larger ones, and then meat is more opaque and its color lighter. In the case of red meats, fast freezing at very low temperatures may yield too pale a color, thus reducing quality.

### VI. NUTRITIONAL VALUE OF FROZEN RED MEAT

Red meats are considered an important source of high-quality food protein, minerals, and B vitamins. Although freezing implies some losses in those compounds (specifically a loss of water-soluble nutrients in the exudate during thawing), the losses generally lack nutritional impact. Freezing can preserve the nutritional value of meat better than most other preservation methods. Vitamin losses are variable depending on the process. In general, the decrease in B vitamin content of frozen meats should be less than 25% for vitamin B1, 15% for B2, 10% for niacin, and 20–50% for B6 or pyridoxine [Mallet, 1994, cited by Genot (4)].

# VII. SHELF LIFE OF RED FROZEN MEAT (CONCLUSIONS)

In terms of quality, freezing is one of the best technologies for preserving red meats. The main limitations on frozen red meat shelf life are the changes in sensorial attributes, specifically color and flavor, due to pigment and lipid oxidation, respectively. Sublimation and drip loss during freezing and thawing may mean a considerable financial loss, but their influence on eating quality seems in reality to be slight (4).

In spite of the extensive literature on the shelf life of frozen meat, experimental data to support that body of theory seem to be scarce and rather old, based on freezing conditions that nowadays are old-fashioned. Furthermore, there is a lack of agreement among data on the storage life of industrial frozen meat (5). Nonetheless, general rules have been established. In this respect, it has been stated that the shelf life of red meat normally ranges from 8 months to 3 years (5), and likewise that beef and lamb can be stored at  $-18^{\circ}$ C for at least 6–12 months and pork for 6 months. Moreover, frozen meat shelf life data, defined as the practical storage life (PSL) recommended by the International Institute of Refrigeration (IIR), are shown in Table 4. In general, PSL seems to be two to five times high quality life (HQL) (4).

Quality problems in frozen meats subjected to extended storage are substantially reduced following some technological indications [specialist packaging, low  $(-18^{\circ}\text{C})$  and constant storage temperature]. Packing/wrapping has a large direct effect on storage life. It can be effective in reducing oxidation, as packaging material can constitute a light and/or oxygen barrier, and dehydration. Fluctuating temperatures during storage are considered to be detrimental to the product.

Although appropriate freezing technology conditions appear to be more important than intrinsic meat quality in extending shelf life, it is nevertheless true that the best way to

 Table 4
 Practical Storage Life Expressed as Months

Product	Storage temperature (°C)		
Product	-12	-18	-24
Beef carcasses	8	15	24
Beef steaks/cuts	8	18 (12) <sup>a</sup>	24
Ground beef	6	10 (10)	15
Veal carcass	6	12	15
Veal steaks/cuts	6	12	15
Lamb carcasses	18	24	>24
Lamb steaks	12	18 (10)	24
Pork carcasses	6	10	15
Pork steaks/cuts	6	10 (6)	15

<sup>&</sup>lt;sup>a</sup> In brackets are expressed the suggested maximum storage times according to Ref. 4.

improve frozen storage life of red meats consists in an integrated package of measures including production, muscle to meat conversion, and freezing technology (INRA).

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## 13

## Chemical and Physical Aspects of Color in Frozen Muscle-Based Foods

J. A. Pérez-Alvarez and J. Fernández-López

Miguel Hernandez University, Orihuela, Spain

M. R. Rosmini

Universidad Nacional del Litoral, Santa Fe, Argentina

#### I. INTRODUCTION

In the mind of the consumer, one of the most important quality attributes of many foods is color (1). It is related to aspects of quality, and disagreement may occur between buyer and seller. It is therefore not surprising that the measurement of color has become an area of much interest and practical importance in recent years (2).

In general, color properties and their measurement are critical quality control parameters. The quality of food products as perceived by humans is very difficult to describe and quantify, as it is usually a function of several food properties (3) including color, which is affected by most of the technological processes involved in the manufacture of foodstuffs (4). Freezing is one such process, and many problems are associated with the maintenance of the color of frozen foods (1). However, in general, little attention has been given to the effects of freezing and frozen storage on the final color of muscle-based food (meat, fish, shellfish, and their products).

Frozen storage is an important preservation method for muscle-based foods. Freezing, in general, offers a long storage life and allows better control of production levels. However, freezing and frozen storage can have marked effects on the structural and chemical properties of muscle foods, including changes in the muscle fibers, lipids, and proteins, all of which have the potential for significantly influencing final quality attributes (3), especially color (5). Quality deteriorates during freezing and frozen storage owing to the osmotic removal of water, protein denaturation, and mechanical damage (6).

The color of muscle-based foods at the retail level exerts a strong influence on consumer purchasing decisions, and appearance probably is the most important factor in determining retail selection. This is particularly true in the case of meat (7). Food stores assign a relatively large display space to meat in order to attract consumers, frequently using bright lights (8). Most of the frozen muscle-based foods are sold in cool or warm fluorescent illumination (9) on a gondola, where different types of processing, e.g., glazing and packaging, are evident. However, to avoid color damage especially in beef, the type of light used should be warm fluorescent light, which shows an excellent color spectrum and

has no adverse effect on meat pigments (10). Some types of product, especially fish and shellfish, are not sufficiently protected from the action of light and oxygen and may show surface discoloration during frozen storage, so that they are frequently packed in vacuum, in gas permeable packages, or in modified atmospheres (11).

#### II. COLOR MEASUREMENT

The color of foods can be defined as the interaction of a light, an object, an observer, and the surroundings of the food.

Objective color measurements may include measurements of several properties or various ratios or color difference indices (12), by summarizing all the reflected colors (wavelengths) and expressing them as one color (13). The color a consumer sees can generally be described in one or two words, which indicate the main color and its shade. However, color measurements, whether descriptive or specific, must be made as carefully as other measurements (12).

The color of foods can be studied in two main ways: chemically, by analyzing the pigments present, or physically, by measuring the interaction of light. Color necessarily requires a light source that illuminates an object, which in turn modifies the light and reflects (or transmits) it to an observer. The observer senses the reflected light, and the combined factors provide the stimulus that the brain converts into our perception of color, a property that has three quantitatively definable dimensions: hue, chroma, and lightness (12).

Several methods are available for objectively measuring the color of foods, some of which depend on the extraction of pigments from food products followed by spectrophotometric determination of pigment concentration (14,15). However, since such pigment extraction methods are time-consuming and tedious, some researchers have sought simpler methods of color measurement. For example, several methods measure the light reflected from the surface of foods. There are also tabulated coefficients of various objective values, which are correlated with panel scores (16). These objective values consist of numerous combinations of percentage reflectance values and tristimulus values such as Hunter Lab, CIE XYZ, Munsell hue, chroma and value or CIELAB. Others researchers have measured reflectance values, which consist of indices and/or differences of reflectance at different wavelengths (17).

#### A. Fundamentals of the CIE 1976 LAB (CIELAB) Color Space

Today, color is be measured by objective methods, with the CIELAB space being the most widely used method because of its degree of international (industrial and research) acceptance (18). It is often used to facilitate the quality control of colored products, including foods, by measuring color differences and characterizing color, so that color quality decisions can be made. This color space has been adopted throughout the world, and many official quality standards for a variety of different products use it, CIELAB replacing other color spaces such as those of Munsell, Hunter Lab (HLab) and CIE 1931 (12).

CIELAB has the advantage of being more perceptually uniform, and it is based on the accepted color description theory that colors cannot be red and green, or yellow and blue, at the same time. Colors are considered as combinations of red and yellow, red and blue, green and yellow, and green and blue. CIELAB is a reasonable descriptor of color (2) and is also used for measuring and ordering an object's color.

The CIELAB space is a mathematical transformation of the colorimetric system first published by the CIE (International Commission on Illumination) in 1931 (19). Although the 1931 system proved useful, its practical application was limited as it did not express differences between colors in a uniform perceptual manner. In CIELAB space, however, the numeric differences between colors agree consistently well with visual perceptions (20). CIELAB color difference equations contain valuable information about the characteristics of human color vision and provide quantitative metrics corresponding to psychological color descriptors (21). Their performance in describing visual color differences is much improved over the tristimulus color space of CIE (19, 22).

CIELAB space was recommended by the CIE in 1976 for use as a color difference metric (2). CIELAB space was specifically recommended for meat color evaluation in 1995 (23), before which time the most used color space was Hunter Lab (HLab), which is still mentioned in some guidelines (12).

Although the studies of this color space were developed for flat two-dimensional objects (21), the CIELAB color space can be visualised as a three dimensional space, where every color can be uniquely located. The location of any color in the space is determined by its color coordinates: L\* (lightness), a\* (the red/green coordinate, with +a\* indicating red and -a\* indicating green), and b\* (the yellow/blue coordinate, with +b\* indicating yellow and -b\* indicating blue). These color coordinates are adimensional (20). The L\*, a\*, and b\* coordinate axes define the three-dimensional CIELAB color space. Thus, if the L\*, a\*, and b\* coordinates are known, then the color is not only described but also located in the space.

Colors in CIELAB can also be described and located using an alternative method, that of specifying their L\*, C\*, and h values (18). The resulting L\*C\*h\* color space is also three-dimensional, where L\* is the same parameter as in CIELAB, while the C\* (chroma) and h (hue) are computed from the a\* and b\* coordinates. L\*C\*h\* notation is preferred, since the concepts of hue and chroma agree well with visual experience (19). Color can also be located using the CIEa\*b\* chromaticity diagram or C\* and h. In both cases, L\* is usually displayed separately, as a number.

One of the most important things that the CIELAB color space describes is the color difference, defined as the distance between the color locations of any two colors in CIE space. This distance can be expressed as DE\* or E\* where DE\* =  $(DL^{*2} + Da^{*2} + Db^{*2})^{1/2}$ , where DL\* is the lightness difference, Da\* is the red/green difference, and Db\* is the yellow/blue difference. It is possible to express DE\* as differences in chroma and hue terminology, instead of Da\* and Db\*, using DE\* =  $(DL^{*2} + DC^{*2} + DH^{*2})^{1/2}$ , where DC\* is the chroma difference, and DH\* the metric hue difference (18).

Experimental knowledge of color difference perception is incomplete, because the CIELAB color space lacks distance uniformity. Color difference perception does not depend only on the measured color tristimulus values of each member of a color difference pair, but also on experimental conditions related to the visual environment, the sample characteristics, and the way in which the samples are presented. These are called parametric effects and include the influence of such factors as illumination, illuminance, surroundings, viewing mode, sample size, sample separation, sample structure, and size of visual color difference (18). These effects are very important for color measurements in muscle foods and must be considered or standardized when preparing the samples for color evaluation (12).

#### B. Color Measurement in Muscle-Based Foods

Measuring the color of muscle-based foods involves two basic methods: human visual appreciation (sensory analysis) and instrumental analysis (absorbance and reflectance methods) (12). Objective determination of color by means of reflectance spectrophotometry is one of the most commonly used methods owing to its close correlation with the visual perception of the human eye. In contrast to absorption spectrophotometry, reflectance is measured over the surface of the object, thus making destruction of the object unnecessary and allowing the changes in color over a period of time to be evaluated (12).

According to the way light interacts with a muscle-based food, it can be classified as translucent or opaque, although such foods rarely fall into one category alone. The color of fresh meat is known to be largely determined by the relative proportions of deoxymyoglobin (DMb), oxymyoglobin (OMb), metmyoglobin (MMb) for fresh meat, and nitrousmyoglobin (NOMb) for cured meat products (24).

Guidelines for color measurements in muscle-based foods are not complete and are only well documented for meat. Fish and shellfish do not have specific guidelines for color measurements, although the guidelines that do exist could be adapted to their specific characteristics. The same basic principles can be used for these types of food when color is measured.

In the standardization of color measurements of fresh meat, the CIELAB color space has been recommended using  $D_{65}$  as light source and  $10^{\circ}$  as standard observer, with the illumination/viewing system as 45/0 or diffuse 8 (d/8) and specular reflectance excluded if within the capabilities of the instrument used (23). In fresh meat, the sampling should be a cross section taken perpendicular to the long axis of the muscle, and the sample should have a minimum thickness of  $1.5\,\mathrm{cm}$  (the sample should be opaque or an opaque backing must be used) (12). In measuring the color of muscle-based food with low myoglobin concentrations, the relationship between sample thickness and light transmittance can be checked with white and black backgrounds. The American Meat Science Association (AMSA) recommended that, in the case of frozen meats, the sample surfaces should be as flat as possible, and that a black rubber gasket slightly larger than the aperture should be used to help level uneven frozen surfaces and block stray light. The history of frozen meat (time, temperature, package, etc) when color is being measured should also be stated, because temperature fluctuations during transportation or storage can affect the color of muscle-based foods.

#### III. OPTICAL PROPERTIES OF MUSCLE-BASED FOODS

From a physical point of view, the color of muscle-based foods is considered a surface phenomenon of an opaque solid, where light falling upon it may undergo processes of absorption, reflection, or scattering, but where, generally, there is little transmission (12). MacDougall (25) described meat color as partly determined by light scattering, presumably due to the gaps existing between myofibrils (26).

Light is unable to penetrate a significant distance into the meat without being scattered (27), such penetration and reflection being pH dependent. Thus a low pH causes greater scattering and a high pH less (28). In frozen muscle foods (meat and fish) it is generally accepted that pH decreases during storage (29, 30), although some authors found that the pH of some foods can also increase during frozen storage (31).

Swatland (32) describes the optical properties of meat as several levels of organization. This author describes four levels. 1, sliding filaments and myofibrilar birefringence; 2, myoglobin and protein precipitation in the sarcoplasm; 3, postmortem fiber shrinkage; and 4, the release of fluid and the macroscopic surface reflectance properties of cut meat. All of these phenomena can affect the color of muscle-based foods in different ways.

Muscle proteins in frozen meat may be affected by factors such as ice crystal formation, dehydration, increased solute concentration, fat hydrolysis and/or oxidation, the presence of atmospheric gases (particularly oxygen), protein oxidation and proteolysis, and rigor temperature. During frozen storage total/myofibrillar protein solubility increases, while the solubility of sarcoplasmic proteins is reduced (33). Sarcoplasmic proteins (in which myoglobin is included) are more susceptible to low-temperature and frozen-storage denaturation than myofibrillar proteins. Pigment also decreased as a result of ice storage (34). Modifications in sarcoplasmic protein fractions during frozen storage occur because endogenous enzymes are released and are not inhibited.

The color of frozen meats is governed by the freezing rate, which affects light scattering properties. Fast freezing results in small crystal sizes, which scatter more light than the large, slow-growing crystals produced during slow freezing. Fast frozen meat is opaque and pale (reflectance of the small ice crystals), and slow frozen meat is translucent and dark (35, 36). Frozen meats in any circumstances can appear darker than fresh meat owing to the concentration of meat pigments during the freezing process (27).

### IV. COLOR PROPERTIES OF MUSCLE-BASED FOODS DURING FREEZING AND FROZEN STORAGE

In general, the quality of frozen muscle-based foods decreases as the storage period increases. Color especially is affected, although other important sensorial characteristics including odor and flavor are also affected (37).

The consumer's acceptance of frozen meat products on display is related to the color of the product: the redder the meat, the higher the degree of consumer acceptance. However, the consumer will reject fish and shellfish when they appear dark. Some authors have used objective parameters to evaluate acceptability as the percentage of reflectance difference between 630 and 580 nm; the a\*/b\* ratio, Lovibond tintometer, red color units, chroma or metmyoglobin content (%), etc. (38, 39).

Different factors affect color and its stability in frozen muscle-based foods. Such factors include the particular freezing technique used (individually quick frozen or IQF, plates, glazing, etc.), temperature (-5 to  $-60^{\circ}$ C), time and storage temperature, storage methods, freezing-thawing cycles, type of packaging, and type and product characteristics (pigment concentration, polyunsaturated fatty acid concentration, etc.) (4, 33, 40-42).

During freezing, enzymes and other components are released. The ice crystals formed may injure the cell and release prooxidants (iron) (41). For any unprotected muscle-based food during frozen storage, surface dehydration may, in extreme cases, produce an important defect known as freeze burn (caused by ice sublimation), which becomes apparent after thawing and affects the appearance and functional properties of the products. This problem is very important for frozen mussels that are sold in bulk without any packaging. Freezer scorch is another defect that can appear during frozen storage. This shows as small gray and white areas, and is also caused by dehydration (27).

The freezing-thawing process is also very important for the physical and physicochemical properties of muscle-based foods. The denaturation of muscle, structural changes, and lipid oxidation induced by the freeze-thaw process affect color. Lipid oxidation is one of the major problems with frozen muscle-based foods (fish, shellfish, and meats). However, the relation between lipid oxidation and pigment oxidation is not fully understood (43).

The primary catalysts of lipid oxidation in skeletal muscles are hemoprotein and iron (44). In general, iron is distributed among five main components: the insoluble fraction, ferritin, hemoglobin, myoglobin, and the low molecular weight fraction (45). The two pools of iron in muscle foods, heme and nonheme, are altered during frozen storage. Nonheme in cod and mackerel muscle increases during frozen storage (46). Since myoglobin and hemoglobin are the pigments responsible for color, this property is affected by the freezing process. The iron released from ferritin may act as a lipid oxidation catalyst in muscle (47). During the freezing—thawing cycle, the distribution of prooxidants and oxidation stability affect color (41). The freezing—thawing process can disrupt muscle cells and leads to the deterioration of subcellular organelles such as mitochondria. Enzymes are released from the mitochondria into the sarcoplasm, while cytochrome c (48, 49) and other heme pigments present in muscle food may also be released and contribute to color loss. These proteins are soluble, so drip loss in muscle may also lead to a less acceptable color.

#### A. Meat and Meat Products

The frozen storage time of muscle-based foods is related to the characteristics of the product. Meat origin (pork, beef, turkey, etc.), type of muscle, pigment concentration, type of pigment, fatty acid profile, antioxidant type and concentration, etc. are related to color retention and discoloration (50–52). Also important to color is the frozen storage time and type of freezing process used.

The effect of packaging on the color of frozen meat is very important especially if one takes into account the relation of meat color and oxygen or other gases such as carbon dioxide. If packaging highly permeable to gas is used, oxygen can come into contact with frozen meat, and different myoglobin states (OMb, MMb) may be formed. As occurs in fresh meat, MMb is formed from OMb (53). Metmyoglobin plays a prooxidant role in fresh and cooked muscle foods, although in fish the oxidation mechanism is still not fully understood. MMb formation increases with the freezing—thawing cycles (41).

Color parameters (CIELAB) and reflectance ratios vary with the different states of myoglobin, while redness decreases as MMb concentration increases (54). Brownness can be observed in packed frozen stored meats (55). In the case of cured meat products, Sakata and coworkers (56) confirmed that in a model system, nitrousmyoglobin, the main pigment responsible for the color of this type of product is stable under frozen storage conditions.

During frozen storage, MMb% values gradually increase (4,55), changing the appearance of muscle-based foods. Farouk and Swan (33) explained this increase by the fact that metmyoglobin-reducing activity decreases during frozen storage, and consequently MMB is accumulated. When meat or fish is treated with carbon monoxide during frozen storage, MMb% values remain constant (4), while meat stored under carbon dioxide is less red than meats stored in other types of packaging.

In general, the color of salted fresh meat (57) and cured products deteriorates (sodium chloride promotes lipid oxidation) more rapidly in oxygen-permeable packaging than in vacuum packaging (58). Bhoyar and coworkers (31) reported that vacuum-packaged restructured meat products scored higher in color and other sensorial attributes

(flavor, juiciness, texture) and showed greater overall acceptability during frozen storage. Vacuum packaging also reduces the amount of MMb in meat (39).

To prevent color deterioration, natural antioxidants (alpha-tocopherol, rosemary extract, ascorbic acid, carnosine, etc.) are frequently added to muscle-based foods (30). The use of antioxidants during frozen storage is particularly useful for low-fat meat products (59). Industrially, other solutions are sometimes used to avoid this problem, one of them being to dip the product in gelatin solutions to create a coating that improves color stability during frozen storage (58). Another method is to dip the product in a 3% brine solution before freezing at  $-45^{\circ}$ C, especially in the case of ascidians (60). In some cases, color changes during frozen storage are related to lipid oxidation, both of which occur at the same time. This is the case of meats with a low myoglobin concentration, but when the myoglobin concentration is high (e.g., in beef), discoloration occurs much earlier than lipid oxidation (50).

When meat is stored in carcass form or as primals, before being cut and packed, the exposure of the meat surface to harmful environmental effects is minimized (61, 62). When large blocks of meat are frozen, color loss is more pronounced on the exterior than in the interior (55).

Thawed pork chops showed better color stability after storage when vitamin E had been added to the feed (63)

From a sensorial point of view, the color shelf life of frozen muscle-based foods depends on species and muscle type (43). The quality characteristics of ovine meat, for example, begin to deteriorate after 180 days of storage. Other attributes also diminish its acceptability to consumers.

A general rule governing the effect of frozen storage on the color of muscle-based foods cannot be provided, because the effect changes according to the product in question and storage conditions. For example, the color of the same sample can vary with the length of storage. For short- and long-term frozen storage, different parameters may act as color indicators. For example, hue may be a better indicator of color in short-term frozen storage (33) while redness is the best indicator during long-term frozen storage.

Objective color parameters may behave differently during frozen storage. Farouk and Price (51) reported that lightness decreased in ovine muscles. Heath and Owens (64) described a similar behavior for chicken meat, while rabbit and bull meat were seen to be darker after frozen storage (40). In the case of lamb chops, brightness increased, and lightness increased in rainbow trout, as a result of the color oxidation pattern of carotenoids. Decreased redness in meat and meat products is caused by a reduction in OMb and DMb concentrations (55), a phenomenon that has been observed in ground pork (51), low-fat ground beef patties (45), rainbow trout, and Atlantic salmon (1, 4). Less information is available concerning yellowness, although it is known to depend on the type of product; for example, in low-fat ground beef patties it decreased, while in rainbow trout and chicken it increased during frozen storage (4, 64, 65).

#### B. Fish and Fish Products

In the case of fish and shellfish, most are caught at sea and are processed immediately, mainly in frozen form, on board ship, to ensure a high degree of freshness.

Fish and fish products are very prone to discoloration. As mentioned above, lipid oxidation takes place, and changes in meat structure or pigment degradation are common. Minces produced from "white" fish have an unacceptable color after six months of frozen storage (66), while "blue" fish products need antioxidants to prevent color changes (30).

To prevent discoloration in tuna (the pigments of which are very prone to color changes), a combined treatment is used. First the meat is frozen in a  $CaCl_2$  solution at -30 to  $-45^{\circ}C$  and then the  $CaCl_2$  treatment is repeated. The freezing process is intensified by using liquid nitrogen during the first 2–3 h of freezing. Flesh color changes and general spoilage can be limited by freezing and storing at -35 to  $-40^{\circ}C$ , while lowering the temperature to -50 to  $-60^{\circ}C$  ensures a high degree of freshness retention over several months' storage.

In some types of fish, it is important to preserve skin color during frozen storage. This can be done by using natural or artificial antioxidants such as tocopherols, ascorbic acid (the most effective skin color preserver) alone or with butylated hydroxytoluene (BHT), and sodium erythorbate. These antioxidants, alone or mixed, significantly improved color retention during the frozen storage of thornyhead rockfish (*Sebastolobus alascanus*) (67).

Fish and shellfish both contain significant levels of polyunsaturated fatty acids. Benjakul and Bauer (41) postulated that the decrease in heme iron due to heme breakdown in fish muscle was affected by freeze—thaw cycles.

Metmyoglobin plays a prooxidant role in fresh and cooked muscle foods. In the case of fish muscle, the oxidation mechanism is still not fully understood, although MMb formation tends to increase with the number of freezing—thawing cycles (41).

It is known that MMb reductase is a component of red blood cells and is found in fish muscles (68). Since some blood is retained in the fillets, the residual activity of this enzyme could result in the retardation of color deterioration (41).

#### 1. Salmonids

It is very important to maintain the color of farmed Salmonids. This can be largely done by dietary means because color is affected by dietary carotenoid pigment. The shelf life or frozen storage stability of these farmed fish are affected by the fatty acid (saturated and unsaturated) profiles and natural antioxidant levels present in the flesh (69). Color studies carried out to date have mainly concerned farmed salmon (*Salmo salar*) and rainbow trout (*Oncorhynchus mykiss*).

The effects of frozen storage are also related to the structural changes that take place during the freezing process, as was mentioned before. However, the changes that take place during frozen storage depend on carotenoid content and stability, characteristics that are mainly related with astaxanthin and canthaxanthin (from natural or synthetic sources) (70).

Salmonid color and its stability depend on storage time and the packaging system. Sheehan and coworkers (1) reported that canthaxanthin-fed Atlantic salmon were more sensitive to frozen storage than astaxanthin-fed fish. For the same storage time (3 months), canthaxanthin levels decreased by 59%, visual color scores and redness values decreasing at the same time, while lightness increased. The same authors reported that no differences in visual color score or carotenoid content were observed in astaxanthin-fed fish.

Salmonoid flesh is mainly dedicated to producing smoked specialities, while freezing affects color. When frozen astaxanthin-fed fish is smoked, the astaxanthin content decreases, while the canthaxanthin content of canthaxanthin-fed fish remains unaffected after 3 months. Canthaxanthin-fed fish are better for smoking (if stored for less than 6 weeks), although when frozen they lose color more rapidly than astaxanthin-fed fish. Nevertheless, Sheehan and coworkers (1) reported that the astaxanthin content depends

on the frozen storage time of the raw material, falling from 9.39 (fresh) to 7.99 after 6 weeks and 7.26 mg/kg after 12 weeks.

Packaging plays an important role in preventing the discoloration of Salmonoid flesh and its processed products. Different packaging materials (69) and techniques are used (vacuum packed, modified atmospheres, etc.), and it is uncommon to find Salmonid flesh in light display cabinets without any protection to avoid discoloration. When flesh (fillets) of rainbow trout is vacuum-packed, astaxanthin or canthaxanthin remains stable (maximum 5% loss) for 6 months at -20 or  $-80^{\circ}$ C (52, 70). When this meat was analysed by color objective methods, no change was observed in redness (a\*), even if a thawing–refreezing cycle had taken place. In general, prolonged frozen storage increases lightness (L\*), redness (a\*), and yellowness (b\*) but decreases the hue (h) values.

Light exposure (mainly fluorescent light) has a catalytic effect on lipid deterioration in frozen salmonoid flesh. Oxygen-impermeable packaging materials inhibit rancidity and increase the length of time the frozen flesh can be exposed to light.

Bjerkeng and Johnsen (69) pointed to the antioxidative effects of astaxanthin during the frozen storage of rainbow trout fillets (70), while the concentrations of dietary vitamin E are also important. The synergistic activity of  $\alpha$ -tocopherol and rosemary oleoresin during refrigerated–frozen storage increased the retention of carotenoids and reduced lipid oxidation.

#### 2. Shellfish

The frozen storage of shellfish decreases overall quality, particularly in the case of shell-on ascidians and mussels. This phenomenon is caused by the development of ice crystals in the muscle, which discolors after thawing. Choon (71) reported that the muscle of shrimps (*Penaeus japonicus*) after 60 days of frozen storage began to blacken, while color sensory scores fell after 7 months of storage (72). The color of the giant Pacific oyster (*Crassostrea gigas*) becomes unacceptable during frozen storage, a problem caused by freezing temperatures rather than glazing in storage.

Shell-on cold-water shrimps (*Pandalus borealis*) packaged in atmospheric air and exposed to light, lose color during frozen storage. In this case the exclusion of  $O_2$  from the pack would improve overall storage stability and extend the shelf life to at least 9 months. Light influences lipid oxidation and color stability, and its exclusion can extend the shelf life by up to 3 months. One of the main problems with shell-on shellfish are shrimp horns, because they can break the packaging materials, and so it is not possible to maintain anoxic conditions (11).

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### 14

### Frozen Meat: Packaging and Quality Control

#### **Alfonso Totosaus**

Universidad Autónoma del Estado de Hidalgo, Hidalgo, Mexico

#### I. INTRODUCTION

One objective of packaging frozen red meat is to preserve the sensorial and functional characteristics of the tissue for as long as possible. Many factors can alter the quality of this product. Among these factors are the intrinsic quality of the meat, i.e., slaughter conditions; the onset, developing, and resolution of rigor mortis; and the aging conditions before freezing. In fact, the freezing procedure is an important factor in the subsequent quality of the product. Packaging material selection is important for its gas permeability, resulting in important quality attributes such as myoglobin oxidation and lipid rancidity in the meat. Finally, handling conditions are important to preserve the quality until the final destination.

Almost a century ago, Clarence Birdseye spent many years on an expedition in Labrador, working for the U.S. Geographic Service. He noticed that the native Americans who lived there put meat pieces into the cold water. In combination with the cold wind and temperature, this resulted in an almost instant freezing of the meat. He also noticed that the taste of those meats were scarcely different from the fresh meats. As a biologist, Birdseye noticed that the quick frozen procedure formed little ice crystals, with no damage to the tissue. When he came back to the United States, after much experimentation, he founded his own company in 1924 developing the Multiplate Quick Frozen Machine (two metallic plates at  $-13^{\circ}$ F, against the low convection tunnel), and offered to the American people quality frozen foods. Since then the frozen meat market had undergone many changes, mainly in the way meat is packaged to extend shelf life and protect it from environmental factors.

Quality control is employed widely in all industrial processes. In the food industry, quality control is especially important because the food components can increase microbial growth and cause human diseases. In the same way, the method applied to preserve and reduce microbial counts in foodstuffs must be subjected to some quality control procedure. In the freezing of red meat, many parameters are important, and they will be discussed in this chapter. It must be emphasized that frozen foods can be safe indefinitely, but the quality usually decreases with the length of frozen storage.

#### II. PACKAGING FROZEN RED MEAT

Packaging of food in general serves to maintain quality and protect hygiene during storage and transport. The choice of suitable packaging materials and in particular of the processing conditions should ensure that as few undesirable influences as possible are brought to bear on the contents. Packaging used to be regarded as a protection against being touched or a means of protecting the hygiene of the product; but now packaging increasingly serves its functional purposes. See-through packs enable the buyer to judge the content visually. Portion packs can help a consumer to avoid concerns over any leftover. Table 1 is a compilation of the principal requirements for a food package (1). The greatest problem may, however, still lie in marketing, overcoming the prejudice against frozen meat or meat that is not bright red. Recent surveys indicate that these obstacles can be overcome (2).

#### A. Before Freezing and Packaging

The most important parameter before freezing the meat is to allow the resolution of rigor mortis in order to avoid cold shortening in the meat itself. This implies that the meat quality had to be the best to permit a longer period of frozen storage through packaging. This assumes also that during thawing the quality losses do not affect the accepted sensorial, physicochemical, or functional properties of the meat.

#### B. Packaging Materials

Consumers may see packaging as an attractive and informative container for fresh and processed meat and poultry. However, because it touches the food, components of the packaging material are considered indirect additives. This is because chemicals in packaging materials can migrate into the food. Meat may not be packaged in a container that is composed of any substances that may adulterate the contents or be injurious to health. All packaging materials in direct contact with food must be safe for their intended use under the Federal Food, Drug and Cosmetic Act (FFDCA). Any material intended for use in food packaging must be formulated in compliance with FDA requirements for its

**Table 1** Requirements for Packaging

Food protection	Protection of the meat from spoilage, drop in quality, damage and loss
Legal aspects	Physiological and hygienic harmlessness according to international and local legislation
Economic aspects	Rationalization and facilitation of food distribution in production of units, storage, dispatching, and selling. Cost-benefit analysis of packaging and performance
Consumer convenience	Information on origin, quality, weight, price, composition, preparation, and shelf life, possibly visual testing the food, easy handling, and convenience. Labeling
Environmental friendliness	Minimum contamination of the environment during manufacture, distribution, use, and disposal, possible recycling

intended use. It must also state the brand name, supplier, and conditions for use, including temperature and other limits (3).

One of the more important catalysts for packaging advancement has been the development of multiplayer plastics. These advancements are partially due to the developments in high-barrier plastics (4). Oxygen permeation is the most important parameter to take into account in the selection of the packaging material.

#### C. Types of Packaging

There are two kinds of packaging: nonpreservative and preservative. In the former, the package protects the product from contamination and water loss without creating in-pack conditions very different from the ambience. Thus, unless microbial growth is prevented by freezing or retarded by chilling, a product in such a pack is highly perishable and has a very short product life. In the latter, preservative packaging has the ability to extend product life by modifying or restricting microbial growth and is achieved by creating and maintaining in-pack conditions that differ markedly from those of the ambient environment (5).

The preservative packaging employed in meat freezing has three kinds of modified packaging conditions restricting microbial growth. The three can be considered as one because all three employ a modification of the surrounding environment: vacuum, modified atmosphere, and gases. The primary function of a meat package, to contain meat and prevent its contamination, is easily accomplished with the range of plastic materials available today. The functions are achieved by creating a new environment for the meat, and the most important change is modification of the gaseous atmosphere. The composition of the atmosphere determines the color of meat and the nature of spoilage that develops (4).

#### 1. Vacuum Packaging

A method widely employed in the meat industry is vacuum packaging to increase shelf life of fresh and frozen meats. Vacuum packaging is a means against chemical breakdown and bacteriological spoilage, because the lack of oxygen inhibits the growth and proliferation of bacteria in the meat (6). Vacuum packaging involves placing a product in a film of low oxygen permeability, removing air from the package, and applying a hermetic seal. The O<sub>2</sub> level is reduced to less than 1% by vacuum packaging. The method has been shown to be effective in inhibiting bacterial growth at low temperatures, resulting in reduced spoilage and extended product shelf life (4).

#### 2. Modified Atmosphere Packaging

Modified atmosphere packaging (MAP) and the more precise controlled atmosphere packaging (CAP) help to preserve foods by replacing some or all of the oxygen in the air inside the package with other gases such as carbon dioxide or nitrogen (3). Modified atmosphere packaging is defined as the enclosure of a food product in gas-barrier materials while providing an environment that has been changed or modified to inhibit the action of spoilage agents. This results in either maintenance of the quality of a perishable food during its natural life or the actual extension of the shelf life (4).

#### 3. Gas Packaging

In gas packaging the product is generally stored in an atmosphere containing an appropriate composition of  $N_2$ ,  $CO_2$ , and  $O_2$ .  $N_2$  is used primarily as a filter to prevent package collapse with foods that absorb  $CO_2$ , or simply to displace  $O_2$  to retard oxidation (4).

#### 4. Skin Packaging

Skin packaging is essentially a modified form of vacuum packaging. A preheated film is dropped onto the product that is supported on a lower web of the same film. The air between the two films is withdrawn and an upper web forms around the food to produce a skintight package that is then heat sealed in a vacuum chamber. It is used primarily for packaging frozen meat products (4).

#### D. Behavior of Meat in the Package

#### 1. Water Loss

In most cases, uncovered meat loses weight by the evaporation of moisture, which dries and darkens the surface. During frozen storage, water is lost from the meat surface by sublimation to colder surfaces in the vicinity. Water is also lost as the principal component of the exudate that inevitably comes from the cut surfaces of chilled meat (2).

#### 2. Tissue Respiration

Meat is a biological material containing respiratory enzyme systems that continue to function after death. Respiration is confined to the surface layer into which oxygen diffuses. The depth of penetration depends on a balance between oxygen concentration at the surface, driving it inwards, and the tissue respiration that consumes the oxygen as it becomes available (2).

#### 3. Microbiological Growth

The surface of a beef carcass may carry between  $10^2$  and  $10^4$  bacteria/cm<sup>2</sup>, and after butchering, joints and pieces of meat for packing are likely to carry considerably higher numbers (2). During the freezing process, the main effect is on water activity. The aqueous portion of meat remains in its liquid phase until reaching its freezing point at some temperatures below  $0^{\circ}$ C (7).

#### 4. Color Changes

Lean meat color derives from muscle pigment, myoglobin, and its reactions with available oxygen. In meat that has been exposed to air for several hours, the penetration depth for oxygen may be 6–7 mm. In the presence of oxygen, myoglobin is oxygenated to oxymyoglobin or oxidized to metamyoglobin. The relative amount of these two pigment forms depends on the partial pressure of oxygen (2).

#### E. Chilled Meat Packaging Requirements

With frozen storage packaging, higher oxygen transmission rates reduce effective product life. Specific customized or proprietary systems may achieve performance specifications using films of higher or lower oxygen transmission rates. There are different types of packaging for frozen meat, depending on the kind of meat to be packaged. Boneless beef and lower value cuts of other species destined for further processing are bulk packed within low-density polyethylene liner bags in cardboard cartons. This package is cheap, provides good moisture barrier properties, is rugged, and still retains film flexibility at temperatures approaching  $-40^{\circ}$ C. However, the high oxygen permeability of the packaging film results in short quality product life because of color deterioration and oxidative rancidity. Polyethylene bags may also be used for frozen carcasses. While oxygen impermeable films are the norm, oxygen permeable films are used sometimes so that the product will bloom. (5)

#### III. QUALITY CONTROL IN PACKAGING FROZEN RED MEAT

Quantity control concentrates on delivering the desired output within the expected delivery date. In this respect, the control function is the action phase of production. Production control serves the dual purpose of directing the implementation of previously planned activities and monitoring their progress to discover and correct irregularities. Plans are converted into action notices that spell out exactly which workers and machines will operate, what the operations will be, and when they must be done. The actions are compared with planned performance to provide the feedback for replanning or initiating corrective actions. (8)

Frozen meat packaging quality control is difficult because there are several factors associated with the meat per se. This means that the meat to be packaged and frozen must be of the best quality possible; it means following good manufacture practices from slaughter, rigor resolution, and meat cutting processes before packaging, plus the adequate frozen procedure to ensure a prolonged and stable shelf life until posterior processes or consumption. The next sections can be considered as control points to obtain a good quality product.

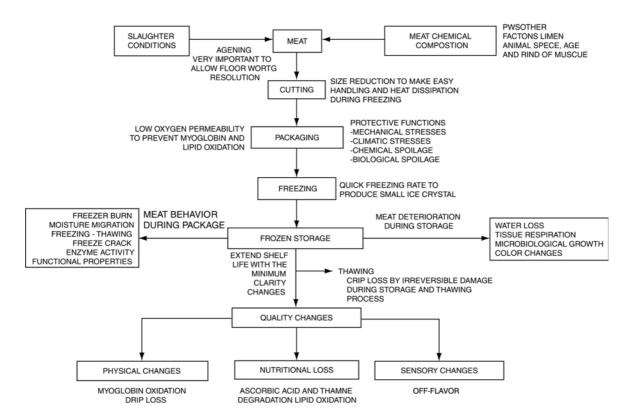
#### A. Quality Changes in Frozen Foods Associated with Packaging

Many changes are associated with packaged meat quality during frozen storage. These changes can be divided into physical and nutritional losses and sensory changes. Physical deterioration is mainly change of color due to oxygen permeability causing myoglobin oxidation, and drip loss by the recrystallization of water, resulting from damage to the cell structure. Nutritional value is affected by oxidative reaction due to moisture escape and the presence of oxygen, causing losses in ascorbic acid and thiamin, and lipid rancidity. The main and more problematic sensorial change is the off-flavor, due mainly to peptide degradation.

#### B. Product Deterioration During Frozen Storage

Freezing and frozen storage produce extensive changes in muscle structural and chemical properties, including changes in proteins, lipids, and muscle fibers, with an overall effect in the meat and meat product quality (9). Many important changes can take place during frozen storage.

Figure 1 is a schematic representation of meat packaging and freezing with factors affecting the overall operation.



**Figure 1** Schematic representation of the quality factors involved in meat packaging and freezing.

#### 1. Freezer Burn

Characterized by changes in the surface appearance of frozen meat or offal, freezer burn is the result of sublimation with the appearance of ice crystals during storage. The desiccation resulting from this sublimation appears as gray patches on the product surface. Fluctuating storage temperatures accelerate the onset of freezer burn. Freezer burn occurs most rapidly at higher storage temperatures when the vapor pressure of the ice in the frozen product exceeds the relative humidity of the air in the freezer (5). Freezer burn can lead to accelerated lipid oxidation. The open structure in severely desiccated meat provides a large surface area for interaction with oxygen. Water loss also causes reactants to be concentrated. Freezer burn can be reduced if the product is suitably packaged in a tight-fitting film that is impermeable to water and vapor, or if the cold storage facility is maintained at a high relative humidity, so-called storage over ice. With meat cuts and offal, freezer burn is most commonly associated with damaged packaging. Loss of pack integrity allows areas of the product surface to be exposed to the external environment. The use of loose packaging can lead to an intrapackaging freezer burn condition manifest by frost or "snow" within the pack (5).

#### 2. Moisture Migration

Moisture migration is the principal physical change in frozen foods and has major effects on the chemical and biochemical properties of frozen foods. It manifests itself in several forms: moisture loss by sublimation, moisture absorption and redistribution in foods or food components, recrystallization of ice, and drip loss during thawing. Moisture loss from foods has the most important economic consequences and has received the most

attention. It is a major factor and sometimes the limiting factor of the shelf life of a foodstuff. The various forms of loss of visual quality are also sometimes referred to as freezer burn. Moisture migration during thawing results in drip, which reduces visual attraction and causes nutrient loss. Apart from appearance, loss of moisture will also affect the food's juiciness and texture. Even when appearance and other quality traits are not affected, moisture migration has a significant quantitative effect in the form of weight loss. Typical weight losses during conventional meat processing, for example, amount to about 1–2% during chilling, 1% during freezing, and about 0.5% per month during storage and transport unless the product is wrapped in an impervious film. On the other hand, some degree of moisture loss is sometimes relied on to ensure food quality and safety. During the chilling of meat, some surface drying is necessary to prevent or retard microbial growth by reducing the surface water activity, as well as to avoid a glassy appearance when the meat is frozen (10).

#### 3. Freezing and Thawing

Drip loss during thawing is caused by irreversible damage during the freezing, storage (recrystallization), and thawing process (10). The main problem with the freezing and thawing process is the formation of large protein aggregates resulting in water displacement, proteins moving closer together, and cross-linking and incomplete rehydration. The last phenomenon occurs because the protein—water affinity is the same or less than the protein—protein affinity (11).

#### 4. Freeze-Cracking

There is an important commercial trade-off between the speed of freezing and the economy of the freezing operation. In general, the cost of a freezing operation increases exponentially with increasing freezing rate; at the same time, the improvement of product quality tends to level off beyond a certain freezing rate. There are two types of freeze-crack: (a) surface-only cracks and (b) cracks originating from inside the product, which then progress to the surface. The most popular explanation has been that mechanical damage induced by cryogenic freezing is due to volumetric changes associated with the water—ice phase transition. In order to retain the best possible quality of foods during freezing, knowledge of the freeze-cracking phenomenon and how to prevent it are necessary. A summary of our current understanding and examples regarding how to predict and prevent freeze-cracking has been presented in this chapter. Additional research on physical properties of frozen foods to model the development of freeze-cracking and detect the likelihood of freeze-cracking is needed (12).

#### 5. Enzyme Activity

Low temperature in meat conservation provokes structural changes in myofibrils during the freezing, due to the remaining activity of enzymes and the interaction between lipids, formaldehyde, and other proteins (11). Storage at low freezing temperatures can slow but not inactivate these enzymes in the tissue. These enzymatic activities in turn may lead to quality deterioration. In meat, the enzymes that remain active are lipolytic (lipases and phospholipases) and proteolytic. Hydrolytic enzymes in frozen foods can result in quality denaturation, and the only way to avoid the consequences of lipolysis in frozen foods is to minimize, through processes involving fast freezing and minimization of ice recrystallization in the product (13), the enzymes from the cellular organelles that house them.

#### 6. Functional Properties

Deterioration in texture, flavor, and color, resulting from biochemical, enzymatic, and functional changes in proteins, however, are problems associated with freezing and subsequent storage at subfreezing temperatures for many fresh and processed foods. Freeze-induced protein denaturation, enzyme inactivation, and related functionality losses are commonly observed in frozen fish, meat, and poultry. Denaturation of protein during freezing and frozen storage can be monitored by measuring alterations in protein surface hydrophobicity, amino acid composition, conformational stability, solubility, aggregation, and enzyme activity. Losses in functional properties are commonly assessed by comparing water-holding ability, viscosity, gelation, emulsification, foaming, and whipping properties. Lipid peroxidation is involved in denaturation and deterioration in functional attributes of muscle proteins during frozen storage. Thus various cryoprotectants (e.g., sugars, sorbitol, and polyols) and antioxidant additives are incorporated into food before freezing to minimize physicochemical changes in proteins and to prevent functionality losses (14).

#### C. Quality Control Charts

The reason for a quality control chart is to determine if the behavior of a certain process maintains an acceptable quality level. Any process has a natural variability, due to minor variations without control. On the other hand, the same process can suffer serious variations in key process parameters. The source of these variations can be assigned to known causes or be due to random circumstances (handwork or machine calibrations). A process is in control when the statistical control only experiences random variations. A control chart can be used to detect the not-random or out-of-control state of a process. It is important that the variation or deviation can be detected quickly to correct the problem (15, 16).

A control diagram or chart is designed to measure certain product attributes, with a central line or average to the process standard and upper and lower control lines where the attribute variations are supposed to be. These limits are chosen so that the natural variation could be attributed to random variations in the process, and beyond or below these limits the process is out of control. With the chart of these variations taken in periodic time intervals we can ensure that the process is under control or that any new problem is taken care of. Apart from measuring the point out of the control limits, the tendency or systematic pattern could be used as an indicator of a serious problem in the production process (16).

In the quality control chart of a process, suppose that  $\mu=50$  and the standard deviation is 0.01. In five hours' sampling the sample average is  $\bar{\mathbf{X}}$ . The limits of  $\bar{\mathbf{X}}$  are based in the standard deviation of the random variable  $\bar{\mathbf{X}}$ . The average of independent observations in a sample size n is  $\sigma_{\bar{\mathbf{X}}} = \sigma/\sqrt{n}$ , and the control limits are determined so that there will be a little probability for a value to be out of the control limits, because the process is in control [it means ( $\mu=50$ )]. With the central limit theorem, under process-controlled conditions, we have  $\bar{\mathbf{X}} \approx N(\mu, \sigma/\sqrt{n})$  or  $\bar{\mathbf{X}} \approx N(50, \sigma/\sqrt{n})$ . As result,  $100(1-\alpha)\%$  of the values of  $\bar{\mathbf{X}}$  are within the limits when the process is in control if we use the limits  $CL = \mu \pm z_{\alpha/2}\sigma/\sqrt{n}$ . It is generally accepted that the control charts for  $\bar{\mathbf{X}}$  are based on the limits referred to as three sigma, or  $z_{\alpha/2}=3$ , meaning  $\mu\pm3\sigma/\sqrt{n}$ . In this way, if we consider the structure of the limits from a hypothesis test point of view, for a determined sample the probability that the  $\bar{\mathbf{X}}$  value will be out of the control limits is 0.0026, because the process is under control. This is also the probability for an erroneous determination that a process is out of control (15). Figures 2 and 3 show a typical control chart and the interpretations of some behaviors, respectively.

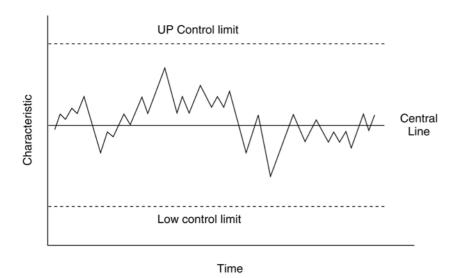
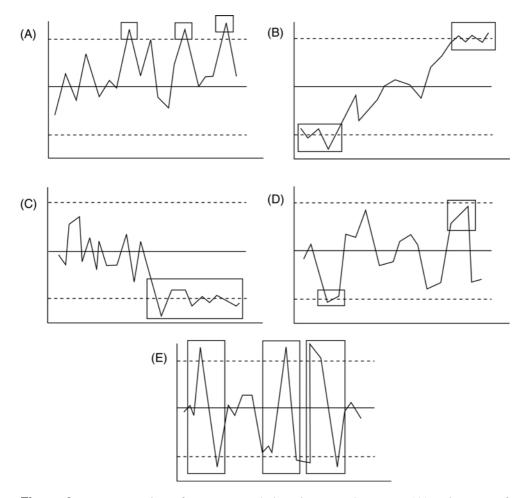


Figure 2 Typical quality chart for control process.



**Figure 3** Interpretation of some control chart in control process. (A) Points out of control: this can be part of a normal process and the causes are easy to recognize. (B) Continuous tendency: increasing or decreasing, depending on the causes of variation. (C) Sudden level change: changes in employees, supervisor, methodologies, standards, or suppliers. (D) Cyclic: tendencies that are associated with the process or process conditions. (E) Instability: erratic points that look very complex, but the sources of variations are normally simple.

#### IV. SUMMARY

Packaging is an important industry operation to facilitate the distribution of meat for retail sale. Packaging by itself needs another unit operation in order to prolong shelf life during storage or distribution. As well, freezing is widely employed because the lower temperature reduces enzymatic and microbial activity in adequate ratio to retard the meat's live tissue respiration. Quality control is useful to control process parameters that can be decisive first in an adequate raw meat quality and second in a stable and prolonged frozen storage.

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# Frozen Poultry: Process Flow, Equipment, Quality, and Packaging

Alma D. Alarcon-Rojo

Universidad Autónoma de Chihuahua, Chihuahua, Mexico

#### I. INTRODUCTION

Foods can be frozen by direct immersion into a refrigerating medium, by indirect contact with a cooling fluid, or in blast freezers. The air blast freezing method is the most widely used in fresh and processed meat.

Freezing is traditionally used for keeping poultry products. It has a number of advantages over other preservation methods, such as thermal processing, since it provides better sensory quality and nutrient retention in the finished product.

Freezing delays microbial growth of microorganisms and slows down changes affecting quality or causing spoilage. Although all available freezing methods are used in poultry processing, the choice of freezing equipment is primarily a matter of cost, and factors such as product quality and operational flexibility are secondary considerations.

#### II. FREEZING METHODS

#### A. Continuous Conveyor-Type Sharp Freezer

This type of freezer uses a continuous conveyor moving the product through a tunnel-like system. Some advantages of this equipment are reduced costs, minimal defrosting, and little effect of loading and unloading on product quality.

After loading the product onto a stainless steel belt, it travels into the tunnel sections, where the high velocity air  $(-35^{\circ}F)$  blasts horizontally from the top of the product surface and under the belt. The horizontal air direction alternates from one section to the next so to achieve even freezing. The production line feeds the freezer-belt directly, and this in turn is discharged by conveyor into the storage room (Tressler et al., 1982).

Freezing chicken chunks in a mechanical system can be less expensive than that experienced in a liquid carbon dioxide or liquid nitrogen freezer. Mechanical systems can be designed to provide similar performance to that of a cryogenic system affecting shrink and quality. The main advantages of this type of freezing are low cost and reduction of undesirable burn or graying of the meat edges.

#### B. Spiral freezer

The spiral freezer is a variant of the belt freezers with variable residence time. It is generally used for deep-chilling of poultry products. The main advantages of this system is to allow continuous in-line food freezing, gentle product handling, and compact design, resulting in lower operation cost, reduced process floor space, minimum product weight loss by exposure to very low air temperatures at all times, and easy access for sanitation and maintenance. Its main application is to freeze chicken parts, chicken patties, chicken nuggets, marinated chicken pieces, and breaded chicken.

The freezer is a continuous in-line belt freezer. Products are evenly fed from the production line directly onto the loading freezer belt. It quickly transports the product into the low-temperature freezing zone. The belt spirals go up or down along the rotating drum until it reaches the top or bottom where the frozen product is gently discharged from the freezer discharge port. Owing to the large belt surface available, products can be frozen in single layers or individually for IQF (individually quick frozen) quality, which offers low operation cost compared to CO<sub>2</sub>, nitrogen, and other freezers. The horizontal airflow assures that the coldest air is continuously in contact with all sides of the product.

#### C. Tunnel Freezer

Tunnel freezers are suited for crust freezing or chilling of tray-packaged parts on a continuous operation or to deep freeze bulk pieces and low grades overnight. This system is designed to freeze or chill any product such as chicken pieces, chicken nuggets, meatballs, fish sticks, raw shrimp, or fish fillets.

The freezer is equipped with two or three conveyor lanes, each lane can be manually speeded up or slowed down to meet any product needs. There is also the option to chill or freeze three types of packaged products simultaneously.

Tunnel freezing of unpacked food has two major drawbacks: product dehydration during freezing, and need of periodical equipment defrosting as a result of product dehydration, resulting in considerable time loss. Multistage tubular freezer design reduces moisture loss, usually associated with ordinary air-blast and tunnel freezers, by keeping a relatively small temperature difference between the refrigerant and the air in contact with the product being frozen, and also by holding a high relative humidity in the recirculated air used for freezing. This freezer is a continuous in-line belt freezer designed to minimize product weight loss and assure gentle handling during the freezing process.

#### D. Air-Blast Freezing

In this method products to be frozen are placed on trays, either in bulk or in packages. Trays are then placed on freezing racks in a low temperature room with cold air blowing over the product.

Tunnel freezing is the most commonly used freezing system. In it a long, slow-moving mesh belt passes through a tunnel fed with cold air. Usually the cold air is introduced into the tunnel at the opposite end from the one where the product to be frozen enters, that is, the airflow is usually counter to the direction of the flow of the product. The temperature of the air is usually between -18 and  $-34^{\circ}$ C, although lower temperatures are sometimes used, whereas air velocity varies. Rapid freezing requires recirculating a rather large volume of air in order to obtain a relatively small rise in the temperature while in

contact with the product. Air velocities range from 100 ft/min to 3500 ft/min. Air-blast freezing has several advantages: it can be applied to bulk or package products, it is adaptable to most products, and it can be operated under a wide range of conditions such as product handling, air temperature and rate of flow, as well as to fluidized bed operations. Labor costs can be very low depending upon the product and its requirements. The main disadvantages are the longer time required if the product is previously packaged, as well as evaporation losses if the product is frozen in bulk.

Air-blast tunnel freezers are built in a number of types, depending on the degree of automation. Automatic continuous tunnels are usually operated in-line and do not require any human handling of the product.

#### E. Freezing by Direct Immersion

Liquid nitrogen freezing is used in the poultry industry owing to its flexibility and reduced space requirements, as well as its initial investment cost. Liquid immersion freezing of poultry has been applied to both chicken and turkey, using ethylene glycol or salt solutions as refrigerants. To prevent the product from coming into contact with the coolant, carcasses are packed in impermeable, heat-shrinkable bags.

Since liquids are good heat conductors, a product can be frozen rapidly by direct immersion in low temperature liquids such as sodium chloride brine and sugar solutions. The advantages of this system are that there is a perfect contact between the refrigerating medium and the product, therefore heat transfer rate is very high, and the resulting frozen product is not a solid block because each piece is a separate unit.

A refrigerant medium, to be suitable for immersion freezing, must be edible and capable of remaining unfrozen at  $-18^{\circ}$ C and slightly below. Solutions of sodium chloride were formerly used, but today liquid nitrogen freezers more rapidly than any other method.

Immersing a food product in liquid nitrogen at  $-196^{\circ}\text{C}$  ( $-320^{\circ}\text{F}$ ) freezes the outer product surface instantaneously. It prevents product sticking and minimizes dehydration, thereby increasing yields while enhancing the taste and the appearance of the products. It results in a product that is 100% IQF, even when the food has a sauce coating or a high content of water.

Crust freezing by immersion in liquid nitrogen is ideal for products that are difficult to quick-freeze individually, such as seafood, vegetables, and fruits, marinated or cooked products, since they have tendency to clump or stick to the belt during the freezing process. IQF using liquid nitrogen enables them to be frozen singly, thus keeping their individual forms, textures, and tastes.

Immersion and spray freezing can be done with liquids, including brine or an alcohol such as propylene glycol. The product must be perfectly protected. Turkeys are usually first wrapped in a plastic film. Contact with the liquid freezing medium gives fairly rapid freezing.

Cryogenic media include liquid nitrogen, liquid air, and liquid or solid carbon dioxide. Of these, liquid nitrogen has had the widest commercial development. The extremely rapid or "instant" freezing that is possible by immersion or spray freezing gives a quality for most products that is nearest to the fresh. Evaporation is minimized, and installation and maintenance costs are relatively low.

It is well known that deep freezing is the best system for long-term food preservation, and it is normally achieved by sharp freezing the products in a very cold room maintained at temperatures in the range of  $+5^{\circ}$  to  $-20^{\circ}$ C. The Gordon Johnson Poultry Equipment

Company in the USA developed the original deep-chill method for prepackaged fresh carcasses. Here, products are packaged and chilled to -2 to  $0^{\circ}$ C. For whole packaged birds, a residence time of 25 min is required in an air freezer operating at  $-40^{\circ}$ C. Deep-chilling is a common practice as a second stage cooling operation (Verkamp, 1989).

#### F. Plate Freezer

Plate freezers are used in the poultry industry for the freezing of cooked, deboned meat, mechanically deboned meat, or minced offal, but the freezing time in this system is not less than in an air-blast tunnel. This system has the similar advantage of heat transfer by conduction. However, food products conduct heat more slowly than water, most plate-frozen foods are in containers, and the plate freezer is more costly to build, install, and operate.

#### III. PACKAGING

Proper packaging helps maintain quality and prevent freezer burn. Even small air pockets can cause freezer burn. The best way to get out most of the air is to plunge the bag into a pan of water to force all the air to the top.

The type of packaging material depends on the type of food being frozen, personal preference, and the types of material readily available. The packaging material should be moisture-vapor resistant, durable, and easy to seal, and it should not become brittle at low temperatures. For highest quality, foods need to be tightly sealed in moisture-vapor resistant materials and then frozen quickly at  $-32^{\circ}$ C or below.

Poultry chunks can be frozen in bulk or in a box by several different methods. Meat is frozen by a method that permits them to remain stationary on a belt or tray, or in a package. Packaged items could be frozen with cryogen, but the potential of instant freezing would be minimized because of the insulating properties of the box and wrapper.

#### IV. QUALITY

The quality and safety of the final product depends on how the product is handled before, during, and after freezing. High-cost freezing methods may be difficult to justify unless there is no alternative to get the desired quality. Freezing does not kill spoilage organisms in food; it simply stops their multiplication. Organisms will continue to grow and multiply after the frozen food has thawed. Therefore, the number of bacteria in and on foods must be held at a minimum before freezing. For this reason, keep food and everything that touches it (hands, equipment, and work surfaces) scrupulously clean (Willenberg and Hughes, 1997).

Freezing does not improve food quality, nor does it destroy nutrients. However, freezing affects the texture, color, juiciness, and flavor of foods. Be sure to start with high quality food.

Once the food is frozen, even slight fluctuations in temperature can cause the food to thaw slightly, resulting in a mushy product. In food freezing processes, the presence of large ice crystals is a serious drawback when a good final quality of the product is desired.

The storage of meat in air-cooled stores results in greater weight loss than those resulting from pipe-cooled stores. If the meat is packed, then the former type of store is preferred (Mendez-Bustabad, 1999).

Losses in freezing occur from evaporation and from actual loss of pieces dropping from conveyors and becoming enmeshed in or stuck to belts, conveyors, elevators, or other machinery. Freezing in packages should avoid most of the losses.

"Shrink" or "dehydration" varies with the vapor pressure between the freezing fluid and the frozen item, with the time required to freeze the product, and with the quality of food introduced into the freezer system.

Considerable work has been done over the years to analyze dehydration in freezing systems. It has been proven that dehydration is minimal in air-blast systems that are properly designed. Well-designed mechanical systems approach cryogenic freezing systems in both dehydration and freezing times, as the vapor pressure differences are lower than cryogenic by about 60%, and the time to freeze approaches that of cryogenic freezing.

In the tunnel freezer the product shrinkage is minimized to as low as 0.15% because of the unique horizontal air flow pattern. It is emphasized that any bulk-freezing system that reduces exposure time will reduce evaporation losses.

Evaporation losses are important to the processor, as they affect the quantity of product he sells. But there is no loss of nutrients. The buyer gets all of them. Drip losses are of no immediate concern to the processor. They are important, however, to the user. Only after the product is thawed do they appear. The method of thawing and cooking may help control drip losses, especially if the cooking of a fish or meat product is begun before it thaws. If we look at these problems as a whole, without concern at any particular midpoint, what is important is the amount and quality of food, after preparation by the user, that is actually served.

Drip losses after thawing are also important in the overall economics. If a freezing system is available that will eliminate or reduce drip losses, it can be evaluated in the same manner as evaporation losses. The glazing of fish fillets seems to offer one way of minimizing evaporation losses. However, textural and other quality changes caused by a poor freezing method cannot be so modified.

Freezer burn usually appears as grayish-white spots on the food surface. Although it toughens food and causes off-flavors, it is not harmful. Freezer burn is not reversible. Always tightly wrap foods in moisture-vapor-proof freezer wrap before freezing. Increased frozen storage time can increase the possibility of freezer burn. Follow recommended storage times—about six months for poultry.

Bacterial cross-contamination of meat during LN immersion freezing can occur, but the use of good sanitation practices and products with low microbial numbers can limit this occurrence (Duckett et al., 1998).

After the product is frozen, its vapor pressure drops radically. In fact, this will occur after only the surface is frozen. The colder the product is before it enters the freezing zone, the lower will be the evaporation losses. Precooling in a moist environment is recommended to minimize evaporation losses.

Poultry to be sold frozen are rapidly frozen in blast freezers. The commercial blast freezer quickly takes the turkey to a freezing temperature, ensuring optimum safety and quality. They are then stored in freezers at  $-32^{\circ}$ C or below. Frozen poultry is transported in refrigerated trucks to their destination.

Freezing to  $-32^{\circ}$ C inactivates any microbes—bacteria, yeasts, or molds—present in food. Once thawed, however, these microbes can again become active, multiplying under the right conditions to levels that can lead to food-borne illness. Since they will then grow

at about the same rate as microorganisms on fresh food, thawed items must be handled as any perishable.

Subzero freezing temperatures can destroy trichina and other parasites. However, very strict government-supervised conditions must be met. It is not recommended to rely on home freezing to destroy trichina. Thorough cooking will destroy all parasites.

Enzyme activity can lead to the deterioration of food quality. Enzymes present in animals, vegetables, and fruit promote chemical reactions, such as ripening. Freezing only slows the enzyme activity that takes place in foods. It does not halt these reactions, which continue after harvesting. Enzyme activity does not harm frozen meats or fish and is neutralized by the acids in frozen fruits (Food Safety and Inspection Service, 1994).

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## Freezing Seafood and Seafood Products: Principles and Applications

**Shann-Tzong Jiang** 

National Taiwan Ocean University, Keelung, Taiwan

**Tung-Ching Lee** 

Rutgers University, New Brunswick, New Jersey, U.S.A.

#### I. INTRODUCTION

Fish and shellfish are perishable and, as a result of a complex series of chemical, physical, bacteriological, and histological changes occurring in muscle, easily spoil after harvesting. These interrelated processes are usually accompanied by the gradual loss or development of different compounds that affect fish quality. The quality changes are highly influenced by many factors, the most important of which is temperature. If fresh fish is not properly stored, exposure to ambient temperature can cause serious deterioration in quality. Commercially, icing or chilling continues to play a major role in slowing down bacterial and enzymatic degradation of fish muscle. However, this process is not designed to eliminate totally the changes in quality, since it only offers protection for 2–3 weeks, depending on the species.

Freezing of food is an excellent method of preservation with wide applications. Freezing inhibits the activity of food spoilage and food-poisoning organisms, and the low storage temperature greatly slows down the enzymatic and biochemical reactions that normally occur in unfrozen foods. Freezing accomplishes these objectives in two ways: by lowering the temperature of the food and by the removal of water by converting it into ice. Lowering the temperature to below the freezing point inhibits the growth and activity of many but not all microorganisms. Converting most of the water into ice with the concomitant increase in concentration of the dissolved substances reduces the water activity of the food to the point where no microorganisms can grow. Although biochemical reactions slow down at lower temperature, they will, unlike microbiological activities, progress even at low commercial freezer storage temperatures. In addition, conversion of water into ice initiates complex physical and physicochemical changes that can cause generally deteriorative quality changes not ordinarily occurring in fresh foods. Prefreezing processing, such as blanching, freezing, and storage conditions, should therefore be selected, individually, for each product to minimize the effect of these deteriorative reactions.

In most foods frozen commercially, water is the major component. Most of the water in the tissue dissolves soluble cell components, while a small part is bound up in hydrates and in macromolecular colloidal complexes.

In addition, much of the aqueous solution is part of the gellike or fiberlike structures in the cell. The most obvious change that occurs on freezing is the solidification of water, which means that water is removed from its normal position within the tissues. It appears that removal of water from its normal position is only partly reversible upon thawing, leading to drip and other changes. Drip is the exudate from thawed tissue, and it is difficult in practice to distinguish it from any superficial moisture or glaze. There is sometimes an apparent enhanced susceptibility to invasion by microorganisms owing to the moist surfaces that occur during thawing. The conversion of water into ice increases the concentration of soluble cell components (in some cases to the point where they become saturated and precipitate), changes the pH of the aqueous solution, and consequently affects the amount of water that is involved in the colloidal complexes and in the gellike and fiberlike structures. The concentration of cell components leads to a high concentration of electrolytes, some of which interpose themselves in the polypeptide chains of proteins, leading to protein denaturation. In living cells this often leads to death (for example, freezing and frozen storage causes a slight reduction in numbers of most microorganisms), but in foods, which usually consist of dead tissue prior to freezing, it can lead, during storage, to irreversible changes in texture (e.g., toughness in fish) and to undesirable biochemical reactions (enzymatically produced off-flavors). An understanding of freezing therefore involves physical, physicochemical, and biochemical aspects.

As mentioned above, freezing is an excellent process for keeping the original quality of foods, such as fish, for long periods of time (commercially, up to 12 months or more). Freezing and subsequent frozen storage are particularly useful in making seasonal species of fish, like herring and mackerel, available all year round. In addition, freezing preservation is also applied in a number of different products made from various fish species. For example, tuna is frozen on board large commercial fishing vessels, brought to land, and then thawed for the canning process. In the production of various value-added fish products, freezing is applied to breaded and battered fish sticks, fillets, steaks, or nuggets. Likewise, high-quality fish are usually filleted, frozen, and eventually sold to consumers.

Ideally, there should be no distinguishable differences between fresh fish and frozen fish after thawing. If kept under appropriate conditions, fish in the frozen state can be stored for several months or more without appreciable changes in quality. However, it is now well recognized that deteriorative changes take place in fish and seafood during freezing, frozen storage, and thawing, which influence the quality of final products. Considerably more knowledge of the basic structure of fish muscle and its chemical composition is essential to understanding these changes that occur during processing.

#### II. NATURE OF FISH MUSCLE

Fish muscle has a unique arrangement of muscle fibers. It is divided into a number of segments called myotomes, which are separated from one another by a thin sheath of connective tissue called the myocomma or myoseptum. The number of myotomes in fish is dependent on the size of fish, while their diameters vary from head to tail (1). There are two major types of fish skeletal muscles, white and red. The red or dark muscle lies along the side of the body next to the skin, particularly along the lateral lines, and may comprise

up to 30% of fish muscle, depending on the species (2). Cells in red or dark muscle contain more lipids than those in white muscle (3,4); they are basically employed for sustained swimming activities, functioning aerobically using lipids for fuel. In addition, red muscle has more mitochondria (5) but less sarcoplasmic reticulum than white muscle (6). It has a large supply of oxygen and a high content of myoglobin, the colored compound that gives its red color. These characteristics, coupled with the presence of the large amount of lipid, particularly among the fatty species, present a serious problem of preservation because of increased susceptibility of this muscle to lipid oxidation. The red muscle of some species has also been reported to contain enzymes that are responsible for chemical reactions such as lipid oxidation and the conversion of trimethylamine oxide (TMAO) to dimethylamine (DMA) and formaldehyde (FA) (7,8). The shape of red muscle area in different species varies considerably. Lean fish such as flounder, hake, sole, cod, pollock, and whiting have a very small amount of red muscle, which lies along the fish skin, whereas the fatty and semifatty fish species have larger areas of red muscle.

White muscle, on the other hand, constitutes the majority of fish muscle. Unlike red muscle, it has minimal myoglobin and a restricted blood supply (5). Often referred to as the fast tissue (9), it is used for anaerobic activities such as short bursts of swimming activity. This muscle exhibits rapid, powerful contractions, the energy for which is produced by reducing glycogen to lactic acid anaerobically (5).

Intermediate between these two types of muscle are intermixed red and white muscle, commonly referred to as "mosaic" muscle (10). In some fish, this is a thin layer of muscle that separates the red from white muscle. However, in other fish, such as salmon, carp, and trout, this muscle is scattered throughout the body of the fish.

The chemical composition of fish varies depending on several factors, such as age, species, gender, maturity, method of catch, fishing grounds, and other seasonal and biological factors. Even within the same species, chemical composition may vary significantly. Generally, fish contain a considerable amount of protein, lipid, and water and small amounts of vitamins and minerals. Other components such as nonprotein nitrogenous compounds are also present in the muscle. These include urea, taurine, peptides, free amino acids, and nucleotides such as inosine and hypoxanthine (11). These compounds, together with the macronutrients found in fish muscle, may be particularly important to fish processors, since they are frequently used as spoilage indices.

#### III. PHYSICAL ASPECTS OF FREEZING

#### A. Formation of Ice

From a physical point of view, fish, land animal, and vegetable tissues can be roughly considered as dilute aqueous solutions. When they are chilled below  $0^{\circ}$ C, ice crystals form at a temperature characteristic of the product and the initial freezing point (FT), which is also the temperature at which the last ice crystals melt on thawing. The freezing point directly depends on the molar concentration of dissolved substances presented, but not on the water content. Fruits, for example, have high water content and a freezing point of -2 to  $-3^{\circ}$ C, while fish contain less water, yet have a freezing point of about -1 to  $-2^{\circ}$ C. The difference is due to the high sugar and acid content in fruits as compared with the low solute content of fish meat. Ice formation occurs during freezing only after a certain degree of supercooling (supercooling is the phenomenon of reducing the temperature of a solution or material below its freezing point without crystallization occurring) has been achieved, and the formation of ice is accompanied by a heating up of the supercooled

product close to the freezing point. In commercial practice, the amount of supercooling is usually insignificant.

As the products are progressively cooled below their initial freezing point, more and more water will be turned into ice and the residual solutions will become more and more concentrated. If, at any time, the products are heated, some of the ice will be turned to water that will then dilute the residual solutions. The ratio of ice to residual solution in frozen foods is a function of temperature and initial concentration of solutes. At a temperature lower than  $-40^{\circ}$ C, there is little or no measurable change in the amount of ice presented in most frozen foods. The percentage ratio of freezing (RF) of frozen foods is usually estimated as follows:

$$RF(\%) = 100 - \left(\frac{FT}{\text{tem. frozen food}} \times 100\right)$$

when RF represents the percentage ratio of freezing and FT represents the freezing point of the frozen food.

#### B. Ice Crystal Size

Once water has started to freeze, the rate of ice formation is a function of the rate of heat removal, as well as of the rate of diffusion of water from the surrounding solutions or gels to the surface of the ice crystals. At slow rates of cooling, few crystallization centers are formed, and the ice crystals grow to a relatively large size. The water in the cell diffuses through the cell wall, leaving the cells in a collapsed condition. Very large ice crystals (few crystals) can lead to mechanical damage to the food product. The cells become physically separated over relatively long distances. As the freezing rate increases, the number of ice crystals increases while their size decreases. Many studies have been done on the effects of size and location of ice crystals on the quality of frozen food. It appears that, for most foods, the size and distribution of ice crystals, encountered in commercial practice, have relatively little effect on organoleptic quality. However, very slow freezing results in undesirable effects like drip on thawing, while very fast freezing may improve the texture of some products.

#### C. Dimensional Changes

The volume change accompanying the conversion of pure water into ice is about 9%. The volume change of foods as a result of ice formation is less, about 6%, because only part of the water present is frozen and because some foods contain spaces. This volume change has to be taken into account in equipment design. In very fast freezing (for example immersion of large items in liquid nitrogen), it can lead to the buildup of excessive pressure inside the product, causing breaking and shattering.

#### D. Completion of Freezing

The freezing process is, for practical purposes, completed when most of the freezable water at the thermal center of the product has been converted into ice, which coincides for most products with the temperature at the thermal center becoming colder than  $-10^{\circ}$ C. Removal of the product from the freezing equipment before this point is reached may

result in slow freezing at the thermal center. It is preferable to freeze the product until the equilibrium temperature (average temperature) is  $-18^{\circ}$ C or colder.

#### E. Desiccation of Frozen Foods

#### 1. During Freezing

It is inevitable that a portion of the water of a product without packaging will evaporate during freezing. The faster the freezing, the smaller the amount of evaporated water. If the product is enclosed in a water-vapor-proof package before freezing, the moisture that escapes from the packet will be nil. But when there is an air gap (of the order of millimeters) between the surface of the product and the internal surface of the package, frost may be deposited inside the package to the extent that moisture evaporates from the product.

For products frozen unpackaged, moisture loss varies from 0.5 to 1.5% or more, depending on the temperature, the rate and method of freezing, and the type of product. The colder the air temperature, the less moisture the air can absorb before it is saturated. Faster freezing methods lower the surface temperature of the product quickly to a value where the rate of moisture evaporation or sublimation is small. Where the surface of a product consists of a moisture-resistant layer (skin of the fish or fat on beef, for example), moisture losses are reduced in comparison with products with cut surfaces (fish fillets, hamburger patties, for example). Proper freezer design for a given product is therefore an important factor in minimizing moisture loss during freezing.

a. During Storage. Moisture loss during frozen storage is a more serious problem because of the length of storage usually involved. Tight-fitting water-vapor-proof packaging avoids all apparent moisture loss. Many frozen foods, however, are still stored unpackaged or packaged in water-vapor-permeable materials. In these cases, moisture loss depends on the average ambient temperature and the temperature of the evaporator, as well as on the temperature fluctuations occurring during storage, and increases with increasing storage temperature, with increasing frequency and amplitude of temperature fluctuations, and with increasing difference between the storage temperature and the lower temperature reached by the evaporator. It should be noted that the saturated vapor pressure of frozen foods is equal to that of pure ice at the same temperature.

If a water-vapor-proof packaging does not fit tightly around the product, desiccation of the product still occurs, but the water removed remains inside the package as frost. The mechanism appears to be as follows:

- 1. The layer of air between product and packaging is subject to temperature variations. As the outside temperature decreases, the temperature of the inside surface of the packaging at a certain moment drops below the product surface temperature, and ice on or in the product will sublime and condense on the inside of the package.
- 2. When the ambient temperature increases, the process is reversed. However, the water vapor will condense on the product surface rather than in the cellular structure from which it evaporated.
- 3. As the cooling—heating cycle recurs, the crystals on the product surface tend to follow package temperature more closely than the mass of the product, and this results in further sublimation of ice from the product.

Frost in packages of frozen foods can amount to several percent of the product weight. Because it leads to ready access of oxygen into the product, frost formation may, with some foods, increase quality deterioration.

The effects of temperature fluctuation depends on the average storage temperature. At warmer storage temperatures, a given temperature fluctuation results in a much larger change in ice vapor pressure than at colder temperatures. As a result the effect of temperature fluctuations on desiccation increases with a warmer storage temperature.

Excessive drying, in addition to leading to undesirable loss of weight, can accelerate oxidative changes by causing the loss of an added glaze, and also by causing the removal of ice from the superficial parts of the frozen products, thus allowing a free access of oxygen to the internal tissues. Some parts of the surface of protein foods may be highly desiccated and their structure even irreversibly deteriorated; light spots known as freezer burn occur on the surface, and the appearance of the produce may become unacceptable. Animal foods (fish, poultry, game) in particular can be affected severely by freezer burn.

#### F. Change in Ice Crystal Size in Frozen Foods During Storage

The changes in shape and size of the ice crystals in frozen foods are caused by periodic variations in temperature experienced during storage; the greater the amplitude of these variations, the greater will be the changes.

#### G. Thermal Radiation in Frozen Food Storage

Thermal radiation has a significant effect on frozen foods in open display cabinets. In these, the top layer of packages may reach a temperature up to 10°C warmer than the average cabinet temperature, leading to quality losses. The temperature of the packages in the top layer represents a thermal equilibrium between the energy transferred by radiation and the energy transferred by conduction and convection. The effect of radiation depends on the emissivity (emissivity characterizes the surface state as far as radiation is concerned. Emissivity reaches a maximum equal to 1 in the case of an ideally absorbing and emitting body [black body] and is zero in the case of a perfect reflector; the latter does not emit or absorb any radiative energy) of the radiating surfaces, e.g., the package and the ceiling or walls. The important radiation is that in the far infrared range (wavelength 8 to 10.10 m) and not that of visible light. A reflecting canopy placed about the open area of the cabinet or packaging in bright metallic foil reduces radiation energy gain markedly. Bright metallic foil packaging may reduce product temperature at the top layer of display cabinets by as much as 6–8°C.

#### IV. PHYSICOCHEMICAL ASPECTS OF FREEZING

#### A. Composition and pH Changes During Freezing

Freezing converts a large proportion of the water present in foods into ice and hence makes the remaining solution more concentrated in dissolved, colloidal and suspended substances. This increased concentration causes a change in acid–base equilibrium (pH) important in the stability of many colloids and suspensions. Shifts in pH (usually toward the acid side) of up to 1 pH unit have been observed under these conditions.

A second result of this increased concentration is the precipitation of salts and other compounds that are only slightly soluble, such as phosphate. This can result in drastic pH

changes (up to 2 pH units) and changes the salt composition of the aqueous solution in foods. These changes often affect the physicochemical systems in food irreversibly. It has been shown, for example, that lactic dehydrogenase, a muscle enzyme, and lipoproteins, important egg yolk constituents, are irreversibly damaged by a pH decrease from 7 to 5 and by increased phosphate concentration during freezing.

## B. Physicochemical Changes in Frozen Foods

Textural properties and the initiation and acceleration of several biochemical reactions depend on the physical chemistry of food constituents and hence are affected by the physicochemical changes brought about by freezing. Loss of water binding properties, resulting in drip, is an example of textural change, while removal of enzymes from cell particles, allowing them free access to substrates in other parts of the cell, is an example of a biochemical reaction initiated and accelerated by freezing. Other physicochemical changes in frozen foods are actomyosin changes in muscle, leading to toughening (fish) or dryness (poultry), loss of turgor in fresh fruits and vegetables, and gelation of egg yolk.

Many physicochemical changes increase with increased salt concentration in the unfrozen phase but will decrease with decreasing temperature as a result of the lower mobility of the salt in the unfrozen phase and the general effect of temperature on chemical reactions. Consequently, physicochemical changes are most damaging in the range between the freezing point of a food and about  $-10^{\circ}$ C. It is important therefore to expose frozen foods for as short a time as possible to this temperature range, both during freezing and during thawing.

## C. Effects of Preparation and Packaging on Frozen Fish

Product preparation and packaging significantly affect the quality and shelf life of frozen fish. If not properly controlled, these processes result in deleterious effects after prolonged storage.

## 1. Product Preparation

Product preparation, in particular, produces a considerable effect on the shelf stability of frozen fish. Whole and eviscerated fish have longer shelf stability than fillets, while minces can usually be stored only for a much shorter period of time. Crawford et al. (12–14) observed this difference during several studies using hake. Minced blocks exhibited reduced quality and accelerated deterioration during storage when compared to intact fillets. This characteristic of minces, which is more apparent among the gadoids, is probably due to the mincing action applied to the fish flesh, which results in tissue damage and subsequently more rapid deterioration. In addition, mixing of red and white muscle during mincing may also result in the dispersion of lipids and some of the enzymes present in the red muscle, leading to greater susceptibility of the minced tissue to deteriorative changes.

#### 2. Packaging Materials and Methods

An efficient packaging system is essential to offset the detrimental quality changes that occur during frozen storage. Packaging materials and methods are obviously designed not only to protect the product from microbial and chemical contamination, dehydration, and physical damage but also to protect the environment from the packaged product. Fish and

seafood can leak gases or unsightly fluids, which may have unpleasant odors. Therefore the choice of appropriate packaging materials and methods for frozen fish is a critical factor in terms of shelf life extension.

Studies have shown that packaging systems affect the quality and shelf stability of frozen fish. For instance, vacuum packaging is well established as a method of providing an oxygen-free environment to minimize the problems associated with lipid oxidation and dehydration during frozen storage.

Several studies have shown the effectiveness of this method for frozen storage of some species of fish. For example, it has been reported that frozen blocks of fillets vacuum packed in moisture-proof films showed high degrees of acceptance and desirable frozen characteristics (12). Likewise, Santos and Regenstein (15) reported the effectiveness of vacuum packaging for inhibiting lipid oxidation in frozen mackerel fillets. Ahvenainen and Malkki (16) examined the influence of packaging on frozen herring fillets stored at different temperatures. They found that vacuum-packed product covered with metallized cardboard had a longer shelf life than a product vacuum packed and stored without cardboard. Vacuum packaging, on the other hand, need not be used if lipid oxidation is not the limiting factor affecting the shelf life of a product. Although the effect or absence of oxygen in packages on some fish species must be considered, other packaging methods such as glazing and the use of heat-sealable packaging films should also be considered. Pacific hake minced blocks stored in moisture-proof, vapor-proof packaging films exhibited superior quality over glazed samples (12). Likewise, Colokoglu and Kundacki (17) observed frozen mullet packed in plastic films with low permeability to oxygen and moisture to have a longer shelf life than when unpacked in the glazed form. However, it should be noted that glazing is still considered to be the cheapest means of protect ing frozen fish during storage and transport. Glazing provides a continuous film or coating that adheres to the frozen product, which retards moisture loss and the rate of oxidation.

Many different glazes are available, including (a) those with inorganic salt solutions of disodium acid phosphate, sodium carbonate, and calcium lactate, (b) alginate solution, otherwise known as the "Protan" glaze, (c) antioxidants such as ascorbic and citric acids, glutamic acid, and monosodium glutamate, and (d) other edible coatings such as corn syrup solids (18). Ice glaze is particularly important in handling frozen fish in developing countries. For products intended for short-term storage, glazing can be practically utilized as a viable alternative to storage without a protective covering. For instance, Jadhav and Magar (19) concluded that glazing was a cheaper alternative to expensive packaging systems for glazed Indian mackerel (*Rastrelliger kanagurta*) stored at  $-20^{\circ}$ C. Glazed samples had a shelf life of 6 months, while samples without a protective covering lasted only 3–4 months.

# 3. Effects of Freezing, Frozen Storage, and Thawing on Color, Appearance, and Consumer Acceptance

One problem encountered during handling, freezing, and storage of fish is the difficulty in retaining the color and appearance of the meat. Changes in color and appearance of fish occur even immediately after catch. Blood pigments become noticeably discolored to various degrees after some period of time. The natural oils in fish play an important role in these color changes. The color of these oils is produced by the colored pigments dissolved in them, which vary from one species to another. These pigments are subjected to considerable oxidation when the fish is frozen and stored. This then results in meat color

darkening to either dark brown or, in some cases, black. This discoloration occurs especially when the fish is stored for an extended period of time. Some fish, like tuna, develop discoloration during frozen storage, reportedly due to oxidation of myoglobin to metmyoglobin in fish blood (20). Other species, such as salmon, swordfish, and shark, also exhibit color changes during storage. Salmon has a pink meat, but when subjected to oxidation its color slowly fades and, in extreme cases, may completely disappear after prolonged storage. Swordfish, on the other hand, develops green discoloration beneath its skin during frozen storage, which according to Tauchiya and Tatsukawa (21) is due to the development of sulf-hemoglobin, a product of oxidation. Shark flesh also discolors and occasionally develops off-odors during storage, most probably as a result of the presence of high amounts of trimethylamine oxide. Interestingly, these marked differences in the color and appearance of frozen fish are quite noticeable in fish sold either as steaks or as fillets, especially when cross-sectional cuts of the fish are made, which permits a comparison of the color of the exposed fish surface with that of the inner portion.

In shrimp, the rapid formation of black pigments, widely known as melanosis, occurs within a few hours after death and is enhanced by exposing the shrimp to air (oxygen). It can occur within just 2–12 hours of exposure. The oxidation reaction leading to the formation of these black pigments can occur at  $0^{\circ}$ C, but at  $-18^{\circ}$ C, no visible spots were detected at up to 3 months of storage (22). Below this temperature, it is believed that melanosis can still possibly occur. It should be noted, however, that although black spots do not necessarily make shrimp unfit for human consumption, such discoloration is usually associated with spoilage, resulting in a decrease in market value. In other shellfish, such as crab and lobster, the development of blue or black discoloration, otherwise known as blueing, is one of the most troublesome problems. Blueing may occur after freezing or during frozen storage, or it may appear after thawing and subsequent air exposure or even shortly after cooking. Needless to say, these changes in color and appearance of fish and shellfish significantly affect consumer acceptance. When consumers select frozen fish, if these products can be seen through the packaging material used, the color and appearance of the frozen product provide an indication as to its degree of quality. As shown in Table 1, undesirable appearance and discoloration of samples have been observed in different frozen whole fish and fillets obtained from Singapore supermarkets (23). Preventing such

**Table 1** Characteristics of Some Frozen Fish Purchased in Singapore Supermarkets

Fish	Thawed-state characteristics
Herring, whole	Skin and meat show rusting
Mackerel, whole	Surface dehydrated, skin and meat show rusting, spongelike meat
Mackerel, whole	Rancid smell in skin and meat
Chinese pompret, whole	Dehydration at lower part of belly and fin
Chinese pompret, whole	Head and belly parts yellowish discolored, spongelike meat
White pompret, whole	Skin and meat show rusting, spongelike meat
Jew fish, whole	Slight rancid smell in skin
Lemon sole, whole	Surface dehydrated, spoiled and rancid smell, spongelike meat
Haddock, fillet	No smell, spongelike meat, cracks
Cod, fillet	No smell, spongelike meat
Flounder, fillet	No smell

Source: Ref. 23.

quality changes is of great commercial importance, since they detract not only from the consumer acceptability of the products but their shelf stability as well.

Thawing also influences the color and appearance of frozen fish and, inevitably, its consumer acceptability. Depending on the thawing technique used, discoloration may occur in fish and other seafood. For instance, when shrimp are thawed at temperatures higher than 0°C, black discoloration or melanosis may occur. This is due to the unnecessary exposure of the shrimp to air, leading to oxidation. A phenomenon known as shimi occurs in frozen—thawed fish meat. Shimi are the undesirable blood spots observed in the belly portion of carp on thawing and are also the distinguishable spots tainting frozen—thawed tuna meat (24). The latter condition is probably due to the blood vessels that remain in unbled tuna meat prior to freezing. When thawed, these blood vessels produce unsightly spots in the meat.

It is possible to determine if the product has been properly thawed and then refrozen. This is particularly noticeable in packaged frozen fish, where spaces on the sides of the package may be filled with a frozen cloudy liquid known as thaw drip. Such muscle drip was originally attributed to the rupturing of cell walls caused by ice crystal formation during freezing, resulting in excess drip during thawing. However, it has been postulated that drip or exudate formation is directly related to the capacity of the fish protein to hold moisture (25). This unsightly exudate from fish muscle indicates, among other things, inappropriate handling, prolonged ice storage prior to freezing, frozen storage at inappropriate cold-storage temperatures, or improper thawing. If not properly controlled, freezing, frozen storage, and thawing generally result in quality changes in fish and seafood that in most cases render the product unacceptable to consumers.

## 4. Effects of Freezing, Frozen Storage, and Thawing on Palatability Attributes

Changes in the texture, odor, and flavor of fish and seafood affect their palatability. Fresh fish have a distinct succulence and a delicate odor and flavor, which are characteristic of the species. These attributes change noticeably when fish is frozen and stored for prolonged periods of time. Interestingly, the changes that influence the palatability of frozen fish and seafood can all be measured organoleptically, and to some extent chemically.

a. Change in texture. Frozen fish gradually loses its juiciness and succulence after freezing and subsequent frozen storage. Such textural changes, reportedly caused by protein denaturation (26–29), are more pronounced in some species of fish, specifically the gadoids. In these species, the chemical breakdown of TMAO to DMA and FA and the subsequent cross-linking of FA to muscle protein (30) produce the textural breakdown in the gadoids and result in a cottony or spongy texture. Fish muscle that has undergone such changes tends to hold its free water loosely like a sponge. When eaten, the fish muscle loses all its moisture during the first bite, and subsequent chewing results in a very dry and cottony texture.

In some species devoid of TMAO-degradation products, muscle fibers also tend to toughen and to become dry during freezing and storage. This is particularly true for most of the nongadoid species and for crab, shrimp, and lobster when stored for prolonged periods. In contrast, the effect of the thawing method on the texture of fish muscle basically depends on the product form. For instance, whole fish, when thawed, exhibits less textural change than filleted fish, basically as a result of the presence of the backbone, which serves as structural support for the flesh. In terms of the effect of the thawing

method, it has been reported that microwave thawing results in higher gel strength of minced samples, when compared to samples thawed under running water (20°C) and samples thawed at room temperature (31). Consequently, the extent of textural changes depends upon the species of fish and upon the conditions of the handling, freezing, duration of frozen storage, and thawing method used.

Several methods have been developed to measure objectively such textural changes, in addition to the gathering of comparative data from sensory evaluations. From texture analysis of minced fish, Borderias et al. (32) concluded that hardness as measured by the Kramer shear cell and puncture (penetration) tests were highly correlated with the sensorial perceived firmness of raw samples, while a compression test was found to be a valuable technique for characterizing the cohesiveness and elasticity of both raw and cooked fish minces.

Alterations in the texture of frozen fish fillets, on the other hand, are difficult to measure objectively, mainly owing to the textural variability that exists within the fish fillets, which is associated with the flakiness and the orientation of the muscle fibers (33). Several attempts have been made to determine the extent of textural changes in fish fillet, including those tested using fish minces (32). However, significant correlations were not obtained.

An instrumental method that may work on fish fillets is the deformation test using the Instron Universal Testing Machine equipped with a flat compression plate. As a nondestructive test (34), it can potentially be modified to conform to the irregular shape and the segmented structural orientation of fish fillets.

b. Changes in Odor and Flavor. Other important changes that affect the palatability of frozen fish include changes in the flavor and odor of fish and seafood. Fish are often described as having a "fishy" odor and flavor. Although the term sounds unpleasant, it can also be used to describe the pleasing taste and odor characteristics of freshly caught fish. Such pleasant, palatable characteristics may be retained as long as the fish are promptly and properly frozen, stored, and thawed. However, the transformation of these attributes to unpleasant and unacceptable traits occurs very rapidly in some fish species, particularly the fatty fish species.

Changes in the delicate flavor of fish and seafood generally occur in three distinct phases during frozen storage: (a) the gradual loss of flavor due to loss or decrease in concentration of some flavor compounds (35, 36), (b) the detection of neutral, bland, or flat flavor, and (c) the development of off-flavors due to the presence of compounds such as the acids and carbonyl compounds that are products of lipid oxidation. These phases, however, only apply to those species with originally delicate, sweet, and meaty flavors. Other species, such as hakes, have an originally bland flavor (35) but develop off-flavors during prolonged frozen storage.

Changes in odor occur in two phases: the loss of characteristic odor and the development of off-odors, which render the frozen product unacceptable. Generally, fish and seafood initially have a fresh, seaweed odor, which can be retained even after freezing and frozen storage. However, gradually such odor is lost, and eventually an unpleasant odor is given off, particularly when there is an inappropriate storage temperature. The development of unpleasant odor is due either to lipid oxidation, a reaction more apparent among the fatty fish species that results in the production of a strong oily, blow oily, or rancid odor, or to the degradation of TMAO, which leads to the production of an unpleasant ammonia odor. Other species such as white hake (*Urophysus tenuis*) initially give off weak odors of sweet boiled milk, but when frozen storage is extended, hake assumes weak off-odors (often described as milk jug odor) followed by a sour milk odor.

#### V. BIOCHEMICAL ASPECTS OF FREEZING

## A. Postmortem Glycolysis and Lipid Oxidation

Fish muscle obtains energy by hydrolyzing adenosine triphosphate (ATP). At any one moment, its concentration is relatively small. During life it is quickly resynthesized using the energy produced when glycogen is oxidized to carbon dioxide and water. On the death of the fish, metabolism in the muscle continues for some time. Postmortem glycolysis, however, is a relatively inefficient process, and it cannot maintain ATP at its in vivo level. Once ATP has fallen to a critical concentration, it can no longer prevent the major proportion of the muscle actin and myosin from cross-linking. This causes the loss of elasticity known as rigor mortis and usually a slow irreversible contraction. The continuing production of lactate and H<sup>+</sup> ions causes the pH of the muscle to fall from its in vivo value of about 7.2 to the so-called ultimate pH, which is usually about 5.5. A pH of 5.5 is near the isoelectric point of the muscle proteins, at which they have minimum water holding capacity and a consequent relatively high tendency to drip on thawing. A higher ultimate pH, therefore, means a greater water holding capacity than a lower pH. The quantity of glycogen present in the muscle at the moment of slaughtering will clearly determine how far the pH will fall during postmortem glycolysis. Like most chemical reactions, postmortem glycolysis is temperature dependent. It is generally found that the lower the temperature at which this process occurs, the slower is its rate. Thus if the carcass is maintained at body temperature after death, the rate of pH fall, of ATP depletion, and of rigor mortis onset is fast. If, however, the muscle is chilled quickly, these changes are slowed down, and the water holding capacity of the muscle remains relatively high.

There is no practical "cold shortening" problem with fish properly chilled after catching. Fish muscle shows the least shrinkage if held at about 0°C. At warmer temperatures the shrinkage and weight loss are greater and they may be quite substantial for a fillet removed from the skeleton prerigor and kept at room temperature. One qualitative difference between fish and meat is the generally lower glycogen content in fish than in meat animals rested before death. Consequently, the post mortem fall of pH in fish is smaller and the resistance against surface bacterial growth less than in meat. In many fish species, therefore, bacterial spoilage is an overwhelming factor.

The fat composition of fish differs markedly from that of meat, fish fat containing a higher proportion of polyunsaturated fatty acids. Although this factor may vary with species and is also influenced by dietary fat intake, it nevertheless implies that fish, in particular fatty fish, are very prone to development of rancidity by autooxidation. Such rancidity may even develop in fatty fish held before freezing, but it is particularly during storage of frozen fish that great care in packaging and in the use of low temperatures are necessary to preserve quality.

## **B.** Denaturation of Muscle Proteins

The proteins of fish muscle differ from those of meat, especially in their higher susceptibility to cold store damage. Frozen storage of fish causes an increase of drip loss on thawing, toughness, coarseness and dryness on cooking, and loss of the desired glossy pellicle on smoking. These changes are highly associated with the so-called protein denaturation caused by freezing and subsequent storage. They are temperature dependent, the maximum rate of development being in the range -1 to  $-5^{\circ}$ C. They are considerably slowed down by colder storage temperatures. Many techniques have been used to measure

these changes, such as extractable protein in salt solutions (ionic strength 0.5–1.0), which has been most widely used. The changes are mostly in the myofibrillar protein of fish muscle. In general, the sarcoplasmic proteins seem to be more stable on freezing and subsequent storage. This kind of "protein denaturation" is associated with the reaction of certain free fatty acids or their oxidized products on the myofibrillar proteins. Recently it has been found that the ultimate pH attained by fish can considerably affect texture. Thus low pH in cod is associated with more pronounced toughness and larger drip loss on thawing.

There is drip loss, some changes in flavor and taste, and an undesirable softening occurring in freeze-thawed fish muscle. When the frozen and thawed fish was cooked, the succulence and water holding capacity greatly decreased, and some undesirable changes in texture such as toughness, coarseness, and dryness occurred. Compared with unfrozen fresh meat, the functional properties such as emulsifying capacity, lipid binding properties, water holding or hydrating capacities and gel forming ability were lower in the frozen stored fish muscle. Most of the studies indicated that denaturation of muscle proteins plays a dominant role in the quality changes of frozen stored fish muscle. The fish muscle proteins have been found to be much less stable than those of beef, pig, and poultry muscles (38). The amount of extractable actomyosin decreased with the duration of storage, while no significant change in sarcoplasmic proteins was observed during frozen storage of cod and other fish (36–38). Since the decrease in soluble actomyosin correlated well with palatability scores, it was proposed that denaturation of actomyosin is the major cause for the decrease in eating quality of frozen fish. Although the change in extractable actomyosin is regarded as the primary criterion of freeze denaturation, it still must be noted that extractability data cannot indicate precisely how much protein is denatured and how much is native. According to previous studies, results from electron microscopic analyses (39), decreases in actomyosin peak (20s-30s) areas on ultracentrifugal analysis (40, 41) and viscosity of soluble actomyosin with duration of storage (41, 42) suggested the aggregation of muscle proteins occurred during frozen storage. In addition to aggregation, dissociation of f-actomyosin into f-actin and myosin also occurred. It appeared that the dissociated F-actin, as thin filaments, became entangled and aggregated and that the dissociated myosin monomers folded into globular form. At advanced stages of freeze denaturation, large masses with diffuse outlines were frequently found, indicating the formation of aggregation complex of actin and myosin (43).

ATPase activity of actomyosin and myosin, another property of myosin related to its contractile function, also decreased with the increase of frozen storage (40–42, 44–47). During frozen storage, changes in isolated actomyosin and myosin have been sought in the number of —SH groups (40–42, 44–47), titratable acid groups (48), and net charge (49), and in the salting-out profiles (50, 51). Connell (52) attributed the insolubilization of frozen stored cod actomyosin to the denaturation of myosin rather than actin. However, isolated carp actin denatured progressively with myosin during frozen storage as was demonstrated by SDS-PAGE (53). During the initial frozen storage, it appears that both myosin and actin undergo denaturation, while denaturation of tropomyosin and troponin was observed during prolonged frozen storage (53).

Decreases in the solubility, viscosity, ATPase activity, and number of SH groups of frozen stored rabbit, mackerel, milkfish, amberfish, tilapia, and trout were observed (44–47, 54, 55). Although Connell (56) ascribed the intramolecular aggregation of muscle proteins to the formation of noncovalent bonds (57) rather than to the formation of disulfide bonds, the involvement of the SH group in the denaturation of muscle proteins during frozen storage has been emphasized by Buttkus (54, 55) and Jiang et al. (44–47).

From the studies thus far reported, the cross-linkage of myosin is ascribed to the formation of disulfide bonds, hydrophobic bonds, and hydrogen bonds during frozen storage. Free SH groups are first oxidized to disulfide bonds. However, only a small decrease was found in the number of free SH groups during frozen storage. Therefore the changes appear to be the result of rearrangements of disulfide bonds from intramolecular to intermolecular through a sulfhydryl–disulfide interchange reaction.

Myofibrils, systematically organized complexes of myofibrillar proteins, undergo structural changes during frozen storage of fish. The most noticeable change is the fusion of the myofibrils, as is illustrated by the cell fragility method (58, 59) and fragmentation into short pieces at the Z-bands (60–63). More recently, studies have been done on the denaturation of enzymes during frozen storage (64, 65). Inactivation of enzymes with a globular molecule was considered to be due to the unfolding of intramolecular structure (66).

Many hypotheses have been proposed to explain the denaturation of muscle proteins (67–70). They include (a) the effect of inorganic salts concentrated into the liquid phase of the frozen system; (b) water activity relations; (c) reactions with lipids; (d) reaction with formaldehyde derived from trimethylamine (in fish); (e) autooxidation; (f) surface effects at the solid–gas interface; (g) effects of heavy metals; and (h) effects of other water-soluble proteins (such as protease). Among these hypotheses, effects of lipids (67–72), formaldehyde (73–77), and gas–solid interface of myofibrillar proteins caused by free fatty acids and/or lipid peroxides must occur during frozen storage. Jarenback and Liljemark have shown by electron microscopy that, in muscle frozen stored with added linoleic and linolenic hydroperoxides, myosin became resistant to extraction with salt solution (78). However, recent studies on isolated muscle protein indicate that proteins undergo denaturation in the absence of lipids, formaldehyde, heavy metals, and watersoluble proteins. Another popular view is the so-called salt-buffer hypothesis, which gives attention to the effects of highly concentrated salt solution in the unfrozen phase of frozen muscle proteins. The concentrated salt solution may denature the proteins (67-72).

One of the most prevalent chemical reactions to occur in fish muscle during freezing and frozen storage is the complex phenomenon of protein denaturation. It has been postulated that the rupturing of different bonds in the native conformation of proteins in frozen fish is followed by side-by-side aggregation of myofibrillar proteins, specifically myosin, brought about by the formation of intermolecular cross-linkages (27, 79). It is also believed that the significant decrease observed in the center-to-center distance between the thick filaments of the A-band of the sarcomere after prolonged frozen storage favors the formation of cross-linkages between molecules and stiffens the fibers (78). Such intermolecular cross-linkages result in aggregation (30), which leads to the formation of high-molecular-weight polymers (80, 81) and subsequent denaturation of myosin during frozen storage.

Several relevant theories on protein denaturation in relation to fish moisture and freezing damage have been formulated. One theory worthy of note is that of protein denaturation being affected by the freezing out of water. The conformation of most native proteins has the hydrophobic side chains buried inside the protein molecule. However, some of these hydrophobic side chains are exposed at the surface of the molecule itself. It has been suggested that the water molecules arrange themselves around these exposed hydrophobic side chain groups so as to minimize the energy of the oil/water interface and, at the same time, act as a highly organized barrier, which mediates the hydrophobic/hydrophilic interactions between protein molecules (81). These

water molecules form a network of hydrogen bonds, which contribute to the stability of the highly organized three-dimensional structure of the proteins. As water molecules freeze out, they migrate to form ice crystals, resulting in the disruption of the organized H-bonding system that stabilizes the protein structure. As the freezing process continues, the hydrophobic as well as the hydrophilic regions of the protein molecules become exposed to a new environment, which may allow the formation of intermolecular cross-linkages (30), either within the same protein molecule, causing deformation of the three-dimensional structure of the protein, or between two adjacent molecules, leading to protein–protein cross-links.

Freezing also concentrates solids, including mineral salts and small organic molecules, within the remaining unfrozen aqueous phase in the cell (82), which results in changes in ionic strength and possibly pH, leading to the denaturation of the protein molecule (83). Love (84) considered this concentrated salt in the unfrozen phase to be the main protein denaturant in the frozen muscle system. If proteins are denatured over time in the presence of concentrated solutes, it is reasonable to believe that longer exposure of protein molecules to these denaturants (e.g., slow freezing) should be avoided. However, further work must be conducted to determine the effect of the rate of freezing on shelf life of frozen fish as related to the solute concentration effect.

Several methods have been established to determine the extent of protein denaturation during frozen storage of fish and seafood. According to Jiang and Lee (85), protein quality is more sensitively reflected by the enzymatic activities in the muscles than by its extractability, since small microstructural changes in protein molecules can cause more alterations in the enzymatic activities than in extractability. For example, the actomyosin Ca ATPase, which measures the activity of myosin, can be used as an index of protein quality. Since this ATPase is capable of hydrolyzing the terminal end phosphate group of ATP to give ADP (68), this particular enzymatic activity can be determined by measuring changes in the amount of inorganic phosphate present in the muscle. The loss of enzymatic activity reflects the extent of freeze damage and alteration of the protein structure in the muscle system. Connell (79) reported a loss in Ca ATPase activity in muscle during frozen storage. In a more recent study using mackerel, Jiang and Lee (85) observed a loss of  $\sim 66\%$  of the original Ca ATPase activity of actomyosin after 6 weeks of storage at  $-20^{\circ}$ C.

Visual examination under a transmission or scanning electron microscope is a powerful technique used in the determination of textural changes in fish muscle due to denaturation. The electron microscopic studies of Matsumoto (30) were able to detect damage to the native structure of the protein: aggregation and an entangled mass were observed. However, results from this technique have to be interpreted cautiously, since the fixing processes of tissue or any tissue section may create artifacts by altering the ultrastructural images or by masking the microchanges in the muscle tissue.

The extent of protein denaturation in frozen fish muscle can also be determined by conducting several tests of protein functionality. The physicochemical properties that affect the behavior of protein molecules during processing are defined as the functional properties of the fish myosystem, which include protein homogenate solubility, emulsifying and water retention properties, gelation, and viscosity (86). The most popular tests to determine the extent of protein denaturation during frozen storage of fish, in relation to its functionality, are determination of the loss in solubility or extractability of proteins and measurement of the water retention properties of the fish muscle system.

## C. Effects of Freezing, Frozen Storage, and Thawing on Nutritional Value

Considerable emphasis has been given to the influence of freezing, frozen storage, and thawing on quality indices such as appearance/color, texture, flavor, odor, and the chemical reactions that accompany such organoleptic changes. Less attention has been given to yet another useful area, i.e., the influence of such treatments on the nutritional value of frozen fish and seafood.

Put simply, considerable attention is given to sensorial perceived attributes, because if consumers reject a frozen product on display, it is not purchased or eaten regardless of its nutritional value. Conversely, if consumers are attracted to a frozen product, they tend to buy it whether it has the needed nutrients or not. However, as the market shifts to the development and merchandising of products to meet the demands of health-conscious consumers, the nutritional value of frozen fish becomes of great importance.

When fish and seafood are frozen, and subsequently stored and thawed, protein denaturation occurs in muscle tissues. As a result, formation of thaw drip becomes apparent and consequently leads to the leaching out of dissolved materials. Likewise, there is an increase in the release of a watery "cook liquor" when the product is heated. Such water losses result in the loss of water-soluble proteins; however, such losses do not result in any measurable decrease in the nutritive value of the protein (87). However, such losses lower the proportion of sarcoplasmic proteins in the fish tissue and may also lead to a small loss of water-soluble vitamins and minerals.

Other quality changes, such as lipid oxidation, can also influence the nutritional value of frozen products. Oxidized fish lipids, such as lipid hydroperoxides, may induce oxidative changes in sulfur-containing proteins, producing significant nutritional losses (88).

## D. Effects of Freezing, Frozen Storage, and Thawing on Intrinsic Chemical Reactions

When frozen fish are subjected to excessively prolonged cold storage at temperatures above  $-30^{\circ}$ C, a series of intrinsic chemical reactions occurs in fish tissues. These reactions include protein denaturation, breakdown of TMAO, and lipid oxidation.

## 1. Breakdown of Trimethylamine Oxide

Quite obviously, protein denaturation during frozen storage produces extensive textural changes and deterioration in fish. These changes are more pronounced in some species of fish, specifically the gadoids, and are related to another intrinsic chemical reaction, the breakdown of TMAO.

TMAO is commonly found in large quantities in marine species of fish. It is believed that these species use TMAO for osmoregulation (89). Among the marine species, the elasmobranches contain more TMAO than the teleosts. Among the teleosts, the gadoids have more TMAO than the flatfish. Except for burbot, freshwater species have a negligible amount of TMAO in their muscles, since they do not take in TMAO in their diet beyond their bodies' nutritional requirements, and they promptly excrete any excess.

After death, TMAO is readily degraded to DMA and FA through a series of reactions. This conversion of TMAO to DMA and FA is typically observed in frozen gadoid species such as cod, hake, haddock, whiting, red hake, and pollock (7).

The presence of air (oxygen) affects DMA and FA formation. It has been suggested that oxygen may actually inhibit the reaction by interacting with metal ions, which otherwise would accelerate the TMAO degradation (90). Lundstrom et al. (91) observed that red hake (*Urophysis chuss*) minces stored in the absence of oxygen showed more rapid DMA and FA formation than red hake fillets stored in air. Likewise, the presence of air (oxygen) in packaged white hake (*Urophysis tenuis*) significantly prolonged the shelf life of the frozen samples (15).

TMAO degradation to DMA and FA was enhanced by the presence of an endogenous enzyme (TMAOase) in the fish tissues, as observed in cod muscles by Amano and Yamada (92). They also found an enzyme in the pyloric ceca of Alaskan pollock (*Pollachius virens*), which was believed to cause DMA and FA formation in this species (93). However, evidence also exists demonstrating that breakdown of TMAO to DMA and FA is nonenzymic in nature (94,95). The breakdown of TMAO, whether enzymatically or nonenzymatically induced, is believed to produce destabilization and aggregation of proteins.

TMAO has been postulated to be responsible for stabilizing proteins against conformational changes and thermal denaturation (96). However, the conversion of TMAO to DMA and FA has been implicated in gadoid textural problems during frozen storage (12, 97, 98).

It has been suggested that TMAO's breakdown product, FA, may produce cross-linking of muscle proteins (30) owing to its high reactivity: FA can covalently bond with various functional groups of proteins, such as the amino, imino, guanido, phenolic, imidazole, and indole residues. This reaction induces both intra- and intermolecular cross-linkages of the molecules, thus producing conformational changes.

However, textural changes may also occur during frozen storage for fish species devoid of the TMAO-enzyme system (99). Such textural changes must then be attributed to another type of mechanism that does not involve the cross-linking of protein molecules due to the presence of FA. Gill et al. (99) reported that the presence of FA in red hake resulted in the covalent cross-linking of troponin and myosin light chains, forming high-molecular-weight aggregates. However, when haddock, a species that does not produce FA, was examined, the same cross-linkages were not found at the molecular level, although textural toughening was observed, but not as pronounced as that in red hake. Based on these observations, they suggested that textural changes in haddock were probably due to secondary bonds, such as hydrogen or electrostatic bonds, and not due to FA cross-links.

Clearly, the presence of FA is not the only factor involved in textural changes during frozen storage. However, with certain species of fish, it appears to be of primary importance.

To determine objectively the extent of textural deterioration due to DMA and FA formation and subsequent reactions, the measurement of DMA content is recommended. Due to the equimolar formation of DMA and FA in fish muscle and the observed high reactivity of FA, DMA content is routinely used as an index. Consequently, the DMA test indirectly measures the FA value in fish muscle. However, use of this test is limited to those species known to produce DMA and FA during frozen storage.

### 2. Lipid Oxidation

Another chemical reaction generally associated with quality changes during freezing, frozen storage, and thawing is lipid oxidation. This phenomenon most commonly occurs in fatty fish and is considered one of the major causes of frozen shelf life reduction.

Lipid oxidation results in the development of a condition described as "oxidative fat rancidity." The extent of oxidation in fish lipids varies with the quantity and the type of lipids in the fish muscle, i.e., fatty species are more prone to oxidation than lean species, and species with more highly unsaturated fatty acids are less stable than the other species. When oxidative rancidity progresses sufficiently, it leads to the development of obvious off-tastes and odors, resulting in reduced shelf life.

Changes in fish lipids may be related to changes in protein during frozen storage. Several reports indicate that the unstable free radical intermediates formed during autoxidation attack the protein molecules, leading to the formation of protein free radicals (88). These protein free radicals may cross-link with other proteins to form protein-protein aggregates and with lipids to form protein-lipid aggregates (7).

Another possible mechanism for reaction between oxidized lipids and proteins occurs through stable oxidation products such as malonaldehyde, propanal, and hexanal (26), which covalently react with specific functional groups on protein side chains, including the —SH group of cysteine, the amino group of lysine, and the *N*-terminal amino group of aspartic acid, tyrosine, methionine, and arginine (11). Such interactions increase the hydrophobicity of proteins, making them less water soluble. Free fatty acids (FFA) formed during autoxidation produce indirect effects on textural degradation by promoting protein denaturation (86). FFA are believed to bind myofibrillar proteins, specifically actomyosin, rendering it unextractable (26, 29). According to Sikorski et al. (29), when the hydrophobic sites of FFA interact with protein molecules, the protein molecules become surrounded with a more hydrophobic environment, which subsequently results in a decrease in protein extractability. This interaction may occur through hydrophilic and hydrophobic forces (29).

Several techniques have been developed to assess the extent of lipid oxidation in fish muscle. The most common techniques include (a) the peroxide value (PV) test, which measures the amount of hydroperoxides or peroxides formed during autoxidation (this test provides only a means for predicting the risk of rancidity development) and (b) the thiobarbituric acid (TBA) test, which measures the amount of malonaldehyde formed upon the decomposition of hydroperoxides during the second stage of oxidative rancidity. Other methods are also undoubtedly available. Therefore the choice of techniques depends on several factors, such as the accuracy required and the availability of equipment.

### VI. MICROBIOLOGY

Most matters of animal and plant origin used as human food are subject to microbiological attack as well as chemical, biochemical, and physical changes. At room temperatures the microbial attack is often so rapid that all the other changes play only a minor role. Microorganisms in all food raw material release enzymes into substrates during their growth. Changes brought about by the activities of these microbial enzymes will alter the odor, flavor, texture, and appearance of the product. Occasionally this is advantageous, but in general it causes deterioration and spoilage. The purpose of preservation, however it is accomplished, is to prolong the storage life of the particular food, and this is done either by killing the microorganisms or by inhibiting their activity and multiplication.

Freezing and subsequent storage will kill some of the microorganisms present in the unfrozen material, but this is a slow and variable process depending, in part, upon the nature of the food. Thus freezing cannot be relied upon to substantially reduce bacterial

contamination present in the foodstuff. The hygienic state of the product before freezing is consequently all-important. Storage at temperatures colder than  $-12^{\circ}$ C inhibits microbial growth and therefore is one effective method of preserving food against microbial spoilage.

Three aspects of microorganisms in frozen foods will be considered.

# A. The Resistance of Microorganisms Against Freezing and Frozen Storage

Some pathogens are more resistant to freezing than are ordinary spoilage organisms. A direct examination for common or expected pathogens should therefore be carried out. Most of the common pathogenic bacteria are gram-negative. This group is more sensitive to freezing, frozen storage, and thawing than are the gram-positive spoilage organisms.

## B. Multiplication of Microorganisms in Frozen Foods

Even when microbial growth is completely inhibited, the frozen product can still deteriorate owing to the activity of the released microbial enzymes, which can still catalyze undesirable biochemical reactions in the food. When the handling of fish before freezing is improper, there is the danger that microorganisms may have released sufficient enzymes and toxin to affect the quality of the frozen product. For example, if lipases are produced before freezing, they can cause marked hydrolysis of fats in fatty fish even when stored at  $-30^{\circ}$ C. If fish has been held at relatively high temperatures before freezing, any pathogens present could multiply and some may produce toxins. The latter will survive freezing and constitute a health hazard. A product destined for freezing should receive the same degree of hygienic handling as that which is to be stored at chill temperature.

Several psychrotrophic microorganisms can multiply at freezing temperatures. In practice, bacterial growth does not occur below temperatures of  $-10^{\circ}$ C. This is probably due to the increasing concentration of soluble salts and organic compounds in the unfrozen water, which will decrease the water activity of this fraction. Only the most drought-resistant microorganisms such as fungi and yeasts can grow in these physiologically very dry substrates, and this is why these organisms are the ones that are still capable of growing at temperatures colder than those at which bacteria can grow. Yeasts are not reported to multiply below  $-12^{\circ}$ C and fungi not below  $-18^{\circ}$ C. It must, however, be noted that microbial growth is extremely slow at these temperatures and for practical purposes can be disregarded below  $-10^{\circ}$ C. During any long retention in the upper freezing range down to  $-10^{\circ}$ C, yeasts or fungi may develop and form visible colonies on the surface of the frozen substrate.

#### C. Microbiology of Thawed Foods

On thawing, frozen foods will spoil almost at the same rate as would be expected from unfrozen products with the same microbial population maintained at similar temperatures. Condensation of moisture on the surface of the product should be avoided, as during thawing it may cause a speeding up of microbial growth.

Pathogenic organisms may grow and produce toxin in food without rendering the food unpalatable. They will be occasionally observed in any food even hygienically prepared. Contamination even with small numbers of pathogenic bacteria during preparation of foods for freezing should therefore be avoided as far as practicable.

Competition between different types of microorganisms is important if food that will be further prepared after thawing is stored in the thawed state before such preparation. A food lacking a normal flora of spoilage organisms, but contaminated with a few pathogens, is more likely to present a health hazard than the same food contaminated to the same degree with normal spoilage flora. In the case of frozen foods allowed to thaw slowly, the psychrotrophic flora are likely to dominate and may so alter the substrate as subsequently to inhibit or slow down the multiplication of any pathogens present when thawing is complete. Packaging exerts little effect on the spoilage pattern; even vacuum packing causes a negligible increase in the growth rate of anaerobes like *Clostridium botulinum* during storage after thawing.

# D. Effects of Freezing, Frozen Storage, and Thawing on Microbiological Quality and Safety

It is readily apparent that spoilage changes in fresh fish occur most commonly as a result of bacterial activity. The species of bacteria vary according to storage temperature. In fish stored in ice, *Alteromonas*, *Achromobacter*, and *Flavobacter* spp. predominate. At temperatures between 35 and 55°C, *Micrococcus* and *Bacillus* spp. constitute the main microflora. Some of these microorganisms produce very active proteolytic enzymes, which produce odor, flavor, and textural problems.

When fish and seafood are frozen, the microorganisms present in their tissues are generally inactivated. Thus during frozen storage, microbiological changes in fish tissue are usually minimal. Microorganisms not destroyed by the freezing process generally do not grow and in some cases die off slowly. Although some microorganisms survive storage at very low temperatures, their activities are suppressed, and bacterial numbers may be considerably reduced if recommended temperatures are maintained (100). The temperature below which microbial growth is considered minimal ranges from -10 to  $-12^{\circ}$ C (101). Microorganisms, however, that survive and remain inactivated during frozen storage resume growth when the fish is thawed and may then lead to microbial spoilage of the thawed product.

Frozen fish are far from sterile and cannot therefore be considered a microbiologically safe product. The microbial activities in fish after thawing depend on the degree of freshness of the raw material, the natural microflora in the fish tissues, and the thawing technique utilized.

#### 1. Stability of Frozen Products

The effects of various freezing conditions on quality and shelf stability of frozen fish and seafood have received considerable attention recently. Studies have dealt with either the stability of the frozen product as related to storage temperature and fluctuation in storage temperature or the effectiveness of food additives in providing shelf stability to frozen products.

a. Effects of Storage Temperature. The apparent effects of storage temperature on shelf life stability of frozen fish are related to protein denaturation and lipid oxidation. The effects of temperature on protein denaturation have been comprehensively studied (102, 37). Maximum denaturation is reported to occur at  $-4^{\circ}$ C in cod muscle (36), while changes in extractable proteins in haddock have been found to be greatest at -2 to  $-6^{\circ}$ C (103).

The rate of lipid oxidation and the accumulation of FFA were observed to increase with temperature (77). In a study using various species of fish, it was observed that maximum production of FFA due to enzymic activities of lipases occurred at -12 to  $-14^{\circ}$ C (104), while the maximum rate of lipid hydrolysis was detected at temperatures just below freezing (105).

Storage at much lower temperatures can therefore prolong the shelf life of frozen fish. For example, cod stored at  $-160^{\circ}$ C showed no detectable deterioration after 6 months of storage (77). Even at -65 and  $-50^{\circ}$ C, frozen samples exhibited very few changes after 9 months of storage. Such observations also suggest that low storage temperatures limit the problems associated with protein denaturation and lipid oxidation during frozen storage.

Several studies have been conducted in an attempt to determine the shelf life of frozen fish at different temperatures and to establish storage temperatures that can minimize quality deterioration in specific groups of fish. Poulter (106) reported that *Rastrelliger brachysoma* (club mackerel) stored at  $-10^{\circ}$ C remained acceptable until the ninth month of storage, whereas samples kept at  $-30^{\circ}$ C were rejected after 12 months of storage. *Scomber scombrus* (Atlantic mackerel) stored unwrapped at  $-18^{\circ}$ C were rejected after 3 months of storage, while samples at  $-26^{\circ}$ C remained acceptable until the sixth month of storage (107). Early rejection of fatty species at relatively low temperatures is reportedly due to the development of rancid flavor and odor. Several studies have also reported the same dependence of shelf-life for different fish species on temperature (15, 108, 109).

Clearly, fish composition has an appreciable effect on shelf life stability of frozen fish. For instance, in a comprehensive study using different fish species, it was found that fatty fish such as mackerel, salmon, herring, sprat, and trout had a shelf life of 2–3 months at  $-18^{\circ}$ C, whereas lean fish such as cod, flounder, haddock, ocean perch, and pollock exhibited storage stability of up to 4 months at the same storage temperature (110).

Based on several studies, it is recommended that those species most susceptible to oxidative rancidity be stored at very low temperatures (at least  $-29^{\circ}$ C) while species less susceptible to rancidity should be stored at temperatures between -18 and  $-23^{\circ}$ C (18). For species with textural problems due to the TMAO breakdown, the storage temperature must be below  $-30^{\circ}$ C

- b. Effects of Fluctuations in Temperature. Fluctuations in storage temperatures affect the shelf life stability of frozen products owing to an increase in the size of the ice crystals formed in fish tissues (26). With slight increases in temperature, small ice crystals melt faster than larger ones, so that when the temperature drops again, the melted ice refreezes around the large ice crystals, forming larger crystals. These large crystals accelerate freezing damage and lead to shorter storage stability.
- c. Use of Food-Grade Additives. The effectiveness of different food-grade additives has also received considerable attention recently. The most commonly used types of additives for fish and seafoods function either as antimicrobial agents or as antioxidants.
- d. Antimicrobial Agents. Additives are commonly used in the food industry to prevent the growth of bacteria, yeast, and mold. The selection of an antimicrobial agent or any combination of agents is rather complicated, especially when dealing with fish. The effectiveness of an antimicrobial agent depends on several factors, such as the moisture content of the product and the presence of other microbial inhibitors like smoke and salt.

Several antimicrobial agents have been tested for fishery products. For instance, the sorbic acid salt potassium sorbate (KS) has been found useful in extending the shelf life of

fresh fish. Studies have demonstrated that KS, when applied as part of the ice, increased ice storage stability of red hake and salmon up to 28 and 24 days, respectively (111, 112). KS, in combination with modified atmosphere packaging (MAP), was also determined to be an effective method for prolonging the shelf life of fresh whole and filleted haddock on ice (113).

The shelf life of fresh fish may also be extended under refrigerated conditions with the use of Fish Plus, which exhibits its preservative effect due to the combined action of components such as citric acid, polyphophates, and potassium sorbate. Citric acid lowers the muscle pH, which consequently creates an optimum environment for potassium sorbate to exhibit its antimicrobial effects. Dipping in Fish Plus has been found to extend the shelf life of lingcod on ice to as much as a week (114). Fish Plus may also be used on frozen fish.

e. Antioxidants. An antioxidant is a substance capable of delaying or retarding the development of rancidity or other flavor deterioration due to oxidation. It is normally used in conjunction with freezing to reduce the rate of autoxidation during frozen storage. Antioxidants delay the development of rancidity either by interfering with the initiation step of the free radical reaction or by interrupting the propagation of the free radical chain reaction (115).

The kinetic of antioxidative action was considered to be that antioxidants act as hydrogen donors or free radical acceptors (AH) and react primarily with  $ROO \cdot$ , not with  $R \cdot$  radicals.

$$ROO \cdot +AH \rightarrow ROOH + A \cdot$$

A low concentration of this chain-breaking antioxidant (AH) can interfere with either chain or initiation, producing nonradical products:

$$A \cdot + ROO \cdot \rightarrow$$
 $A \cdot + A \cdot \rightarrow$ 
nonradical products

Different versions of the antioxidant mechanism have been suggested by different authors (100, 115).

Other antioxidants may function as metal-complexing agents, which partly deactivate the trace metals, often present as salts of fatty acid (100), which would otherwise promote the oxidative reaction. Citric, phosphoric, ascorbic, and erythorbic (isoascorbic) acids are typical metal-chelating agents.

Among these antioxidants, erythorbic acid was used in studies by Kelleher et al. (116) and Licciardello et al. (35) of shelf life stability of frozen fish. This antioxidant was emphasized owing to encouraging results with the use of its salt, sodium erythorbate, in retarding oxidation in whiting, chub mackerel, and white bass fillets (35, 117, 118). Licciardello et al. (35) demonstrated the effectiveness of erythorbic acid in the retardation of oxidative rancidity in fillet blocks of Argentine hake stored at  $-18^{\circ}$ C.

However, the use of erythorbic acid is limited to fish species in which rancidity is the main problem. Kelleher et al. (116) demonstrated the effect of this compound on the frozen storage of red hake (*Urophysis chuss*), a gadoid species, in which lipid oxidation is not the limiting factor for shelf life extension. They found that the rate of DMA formation at  $-18^{\circ}$ C in samples dipped in erythorbate solution was significantly greater than the rate in untreated samples. Such an effect of erythorbic acid on DMA formation may be explained by the fact that this acid acts as an alternative and preferred scavenger of

oxygen, leaving metal ions that would otherwise bind to oxygen, and be inactivated, available to catalyze the degradation of TMAO to DMA and FA (119).

#### VII. HYGIENE IN THE PREPARATION OF FROZEN SEAFOOD

Most foods and food products are susceptible to attack by microorganisms, and they are always contaminated by a variety of such organisms present in the food production chain. Foodstuffs are subjected to further contamination during preparation for freezing as a result of contact with the hands of factory staff during preparation, packaging, and transport, and with air or water.

In view of the hazards to health and the effect of microbial contamination on quality, every effort must be made to reduce such contamination to a reasonable level during the preparation of foodstuffs for freezing. Throughout the world, frozen foods have very seldom been the cause of food poisoning incidents.

#### A. Human Contamination

As pathogenic bacteria like Salmonella and Staphylococcus frequently derive from human sources, it is of vital importance that factory employees be aware of the basic concepts of good personal hygiene—the need for frequent washing and the wearing of clean clothes, overalls, hair coverings, etc. The use of rubber gloves and a protective mask for the mouth may be desirable in some instances. When gloves are worn they must be thoroughly cleaned and inspected before use. Medical supervision is advisable and in some cases should be made compulsory. Notices in lavatories should draw attention to personal hygiene, especially, the need for hand washing. Soaps, hand creams, or dips containing antiseptic agents should be readily available.

#### B. Buildings and Equipment

The design of the building should ensure that both buildings and drains are vermin proof. Interior walls, floors, and ceilings should be finished with a nonflaking surface capable of withstanding detergents and sanitizers. All corners should be rounded to facilitate cleaning. The building should be large enough to house production equipment so that all sides of the equipment are accessible for cleaning.

Entrance to the process area should be supplied with adequate washing facilities with foot-operated taps. Wood, which is almost impossible to sanitize, should not be used in contact with food. All windows should be both bird and fly proof.

Equipment usually becomes soiled with organic residues, which act as carriers of microorganisms. It should be designed and constructed to prevent hygienic hazard and permit easy and thorough cleaning. All surfaces should have a smooth, hard, waterproof finish. Cutting boards should be of a hard material; plastic is preferable to wood. Cleaning and disinfection of the food handling area, including equipment and utensils, should be carried out at frequent and regular intervals. Waste materials should be frequently removed, in covered containers, from the working area during factory operation. Processes should be so separated—either in space or in time—as to avoid recontamination of products in which the "bacterial load has already been reduced." For cleaning purposes an ample supply of potable water should be available. Chlorinated water is effective both for in-plant use (when concentrations of around 5 to 10 ppm are appropriate) and for

sanitation of equipment and surfaces (when concentrations of around 100 to 200 ppm are used), but this should be followed by a rinse. Organic residues inactivate chlorine, which therefore should only be used to sterilize already clean surfaces. Chlorine can be responsible for flavor loss or taint (due to the formation of chloramines or chlorophenols). Removal of the chlorine (by thiosulphate addition, often combined with treatment in activated carbon towers) is practiced, especially in ice cream producing plants.

#### C. Cross-Contamination

During the preparation of food for freezing, all efforts should be taken to avoid a buildup of an undesirable microbial population. For some foods, handling should take place at subambient temperature in temperature-regulated rooms, and where a heat treatment is a part of the processing. This should be so severe that most of the microorganisms are killed. After heat treatment, the food should be promptly cooled to avoid multiplication of the surviving bacteria in the critical zone between 50 and 10°C. Cooling water, if used, should be chlorinated.

## D. Bacteriological Control

Proper organization of the various processes from the hygienic point of view is essential, and a constant watch should be kept for lapses in hygiene. This should include a bacteriological control of the various stages in the processing line. Bacteriological methods are now available that give a good estimate of the bacterial load of the raw material and of food contact surfaces. Methods that give a rapid result are especially useful, as they supply plant management with information on the bacteriological state of products actually under preparation.

The results of the examinations should be shown to the factory staff in order to make them comprehend the vital importance of hygiene in food production. Preferably, courses in food hygiene should be held at regular intervals for employees. The aim should be to give those engaged in food production a thorough understanding of the hazard involved.

The above considerations concerning personal hygiene, equipment, and preparation of foods also applies to handling of frozen foods in catering establishments. Thawing of frozen foods in these establishments should be completed as quickly as possible, and any storage of the food after thawing should be in a refrigerator.

#### VIII. PACKAGING

## A. General Requirements

Not only must packaging used for frozen foods meet all the requirements of normal packaging but it must also meet requirements of packaging suitable for food such as

Chemical inertness and stability

Freedom from taint and odor

Freedom from toxic materials that may migrate into the food

Impermeability, or nearly so, to water vapor and other volatile constituents as well as to any odors from the surroundings

Suitability for use in automatic packaging systems

Suitable size and shape for display in retail cabinets

Protection from bacterial contamination and filth

Ease of opening

An attractive appearance

In addition to these general requirements for food packages, frozen food packages should also

Be of such shape as to allow rapid freezing except for I.Q.F. (individually quick frozen)

Permit volume expansion in the freezing process

Be impermeable to liquids and have good wet strength and resistance to water and weak acid

Be able to withstand low temperatures, not becoming excessively fragile at cold temperatures encountered during the freezing process

Not adhere to the contents in the frozen condition

Have a high reflectivity to reduce heat gain by radiation during display in retail cabinets

Be impervious to light as far as practicable

Surround the product closely, leaving the minimum of air entrapped, thus limiting sublimation during storage

## B. Packaging Materials

A wide variety of materials have been used in devising packaging systems for various frozen foods, e.g., tinplate, paper, paperboard with a wax or plastic coating, aluminum foil, plastic film, thermoformed plastics, and laminated combinations of these materials.

Low permeability to water vapor is an important characteristic of packaging materials for frozen foods. Table 2 below compares the permeability of commercially available packaging materials.

#### 1. Paperboard Packages

A paperboard package for foods is generally in the form of a folded carton, either directly printed or provided with printed wrappers on the outside and in some cases with plastic coated liners. The following coating or laminating materials are commonly used:

**Table 2** Water Vapor Permeability<sup>a</sup>

Film type, 0.025 mm (1 mil)	Transmission rate at 38°C (100°F), 90% R.H
Polyvinylchloride	$120-190 \text{ g/m}^2 \cdot 24 \text{ h}$
Polyamid (Nylon)	$120\mathrm{g/m^2\cdot24h}$
Polyester (Mylar)	$25\mathrm{g/m^2\cdot24h}$
Polyethylene, low density	$19\mathrm{g/m^2\cdot24h}$
Polyethylene, high density	$6 \mathrm{g/m^2 \cdot 24 h}$
Cellophane MST-type	$5-23 \text{ g/m}^2 \cdot 24 \text{ h}$
Polyvinylidene chloride (Saran)	$1.5-5 \mathrm{g/m^2} \cdot 24 \mathrm{h}$

<sup>&</sup>lt;sup>a</sup> The data given in this table refer to a test temperature of  $38^{\circ}$ C and the ranking order between different materials in respect to water vapor permeability. Water vapor permeability of a good package should not exceed about  $0.2-0.5\,\mathrm{g/m^2}\cdot24\,\mathrm{h}$  at  $-20^{\circ}$ C and 75% RH. The important factor, however, is not just the water vapor permeability of the packaging material, but that of the complete package.

Wax blends (paraffin and microcrystalline compositions)
Plastics, e.g., polyethylene or polypropylene (on one or both sides)
Aluminum foil

## 2. Wrappers and Bags

Materials most commonly used are waxed paper, hot melt or plastic coated paper, aluminum foil, coated cellulose films such as MSAT (moisture-proof, scalable, anchored, and transparent), and plastic films, such as polyethylene (PE), polypropylene (PP), and polyvinylidene chloride (PVDC). Also of importance are the laminated materials built up from two or more of these materials or other films. Common combinations are cellulose and PE films, sometimes with a PVDC coating.

These materials are used as over wraps, as liners, and as single- or double-wall bags. The bags either are of the prefabricated type or are formed from roll stock on a filling machine.

Particularly important are shrink-wrap materials because of their ability to adhere close to the product, leaving few if any air pockets. Shrink-wrap bags require evacuation of entrapped air before shrinking. Some of these bags can withstand boiling water, so the package can be used for end-cooking of the product before serving.

#### 3. Wooden Boxes

Often used for fish, they require an inner liner or glaze on the product to guard against desiccation.

## 4. Rigid Aluminum Foil Packaging

These packages, in the forms of trays, dishes, and cups, are generally covered with a crimped-on aluminum sheet or a sheet of aluminum foil laminated to paperboard. Normally used for prepared foods and pastries, etc., they allow rapid heating of the product in the package before serving.

## 5. Semirigid Plastic Packages

These are mostly manufactured from high density PE or PP in the forms of trays and plates, covered by lids; as with aluminum foil these can also be used for prepared foods requiring heating before serving, providing only gentle heat, such as steaming, is employed.

## 6. Tin and Composite Containers

These are used mainly for frozen juices, which often have a mobile liquid phase even at cold storage temperatures. A more recent development is to use coated paperboard in the body and aluminum for the ends, coupled with an easy opening device.

#### 7. Shipping Containers

These are normally manufactured from different materials such as corrugated fiberboard or vulcanized fiberboard paper, and plastics. They are often good heat insulators.

## C. Packaging Machinery/Packaging Systems

An essential requirement of any package used in modern industry is that it should form a part of a system that enables the packages to be formed, filled, sealed and handled mechanically on an integrated packaging line.

#### 1. Form-Fill-Seal Machines

These machines form pouch-shaped or tray-shaped packages from heat scalable plastic films or laminates or plastic coated papers in roll form. The packages are formed and filled in the machine simultaneously or consecutively; these machines can work either in a vertical or a horizontal plane.

## 2. Cartoning System

These can be top filled, end filled, or side filled, but irrespective of these differences each machine should perform the operations of erecting, filling, and sealing.

## 3. Shrink Film Wrapping Equipment

These machines apply shrink film materials from rolls round a given number of consumersized packages to form a unit, often replacing cases or boxes. After the application of the film, mostly in the form of a sleeve wider than the width of the contents, the unit is passed quickly through a hot air oven that shrinks the film tightly around the unit.

#### IX. BULK PACKAGING

The practice of storing frozen food in bulk has increased considerably for the following reasons:

The economy of storage space thus achieved

The ability to separate an intricate labor-intensive further processing packaging operation from the essential processing and freezing operation carried out when the raw material is available

Flexibility in final package sizes of various produces

Bulk packaging is extensively applied to fruit and vegetables and to a lesser extent to meat, fish, and poultry. Individually frozen products, the form of freezing particularly applied to vegetables, is most suitable for bulk storage. Products may also be bulk frozen into blocks, this method is used primarily for fish and meat.

After freezing, vegetables such as peas, corn, beans, and sprouts can be stored on site in silos holding one or more hundreds of tons. Bins of corrugated board, metal, or timber construction, with polyethylene liners providing a moisture vapor barrier and protection against dirt, can be used for storage or shipping. Smaller containers may be multiwall paper sacks, corrugated boxes, or fiber drums provided with polyethylene liners. Storage temperatures should be constant to prevent formation of clumps that have to be removed during repacking. This operation normally consists of tipping the contents out of the bulk container and breaking any clumps before passing the product over a screen or through an air cleaner to remove small pieces prior to visual inspection and repacking, care being exercised over the hygiene requirements of this operation.

Frozen-at-sea whole fish is frozen in blocks or individually. In both cases, the fish, if not packaged, should be glazed prior to storage to minimize desiccation. Some fish is slabbed as skin-on fillets or made into blocks of skinned and boned fish for later cutting; both packs should be cartoned or bagged to minimize desiccation.

#### X. FREEZING

Freezing is simply the crystallization of ice in muscle tissue and includes the consecutive processes of nucleation and growth. These processes are central to the effects of different freezing rates and to subsequent effects on meat quality. Meat does not freeze immediately when its temperature drops below the freezing point and the latent heat (i.e., heat required during the phase change during crystal formation) has been removed. In other words there is a degree of *supercooling*. The greater the supercooling, the greater the number of nuclei formed. The number of nuclei is greatest in the extracellular space, and they are only formed within the cell when the rate of heat removal is higher. As soon as the nuclei form, they begin to grow by the accumulation of molecules at the solid/liquid interface. However, the way they grow depends on the microgeometry and the temperature distribution ahead of the freezing front, in a complex way, as a consequence of dendrite formation with supercooling in front of the dendrite growth. An important concept is characteristic freezing time, which is a measure of the local freezing rate and is defined as the time during which the point under consideration decreases from  $-1^{\circ}$ C (freezing commences) to  $-7^{\circ}$ C (when 80% of the water is frozen). The growth of extracellular ice crystals also takes place at the expense of intracellular water. This leads to partial dehydration of the muscle fibers and subsequent distortion. At high characteristic times (slow freezing), the ice crystals are larger and the tissue distortion is greater.

The freezing process can rapidly minimize physical, biochemical, and microbiological changes in the food. This preservative effect is maintained by subsequent storage of the frozen food at a sufficiently cold temperature.

## A. Freezing Process

The freezing process can be divided into three stages:

- Stage 1. This is cooling down from the initial temperature of the product to the temperature at which freezing begins. It must be borne in mind that the act of placing a product in a freezing apparatus does not render it "safe." Time will elapse before warm food passes out of the microbiologically hazardous temperature zone; this is particularly the case if freezing is carried out in slowly moving air as in the freezing in bulk of products such as berries for subsequent jam manufacture.
- Stage 2. This step covers the formation of ice in the products and extends from the initial freezing point to a temperature about 5°C colder at the center of the product. The major part of the freezable water will be converted to ice, and this quite small reduction in temperature is accompanied by a massive enthalpy change.
- Stage 3. Cooling down to the ultimate temperature for storage. When leaving the freezer, the frozen product will have a nonuniform temperature distribution, warmer in the center and coldest at the surfaces. Its average temperature will correspond to the value reached when the temperature of the product is allowed to equalize. In general, it is recommended to cool the product in the freezer to an equilibrium temperature of  $-18^{\circ}$ C or colder. Product leaving the freezer with a warmer temperature will be stored for some

time in relatively unfavorable conditions. Cooling down to storage temperature may take days or weeks.

## B. Freezing Time

The effective freezing time is determined not only by the initial and final temperature of the product and its change in enthalpy but also by the temperature of the heat transfer medium. The dimensions (especially the thickness) and shape of the product unit affect the overall heat transfer, which includes the surface heat transfer coefficient  $\alpha$  and the heat conductivity  $\lambda$  characteristic of the product. When freezing by air blast  $\alpha$  depends on the air velocity and the shape of the product. In an air blast freezer, the rate of freezing increases with increasing air velocity to an optimum value. The refrigeration load necessary to remove the heat produced by the fans increases with the cube of the air velocity; this factor should be taken into consideration when designing air blast freezers. It is important to direct the air circulation in such a way that all product is equally exposed to the air current. In packaged food the packaging material presents a resistance to the heat transfer, depending on its thickness and conductivity. This resistance is increased considerably if air is trapped between the package and the product.

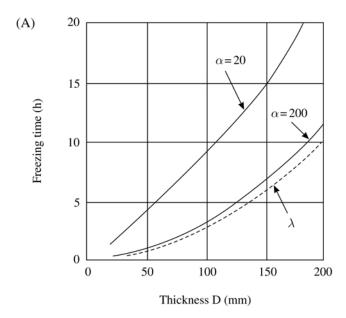
The freezing time as a function of the thickness of fish fillets in packages frozen in a plate freezer ( $\alpha = 200 \, \text{kcal/m}^{2}$ °C including the packaging) and in a blast freezer with medium air velocity ( $\alpha = 20 \, \text{kcal/m}^{2}$ °C) with heat removed from both sides of the package is shown in Figure 1A (120). This graph indicates that during plate freezing the heat conductivity of the product is the main factor determining freezing time; it also shows that during air freezing the heat resistance of the surface (including packaging material) plays a dominant role for the product thickness encountered in practice.

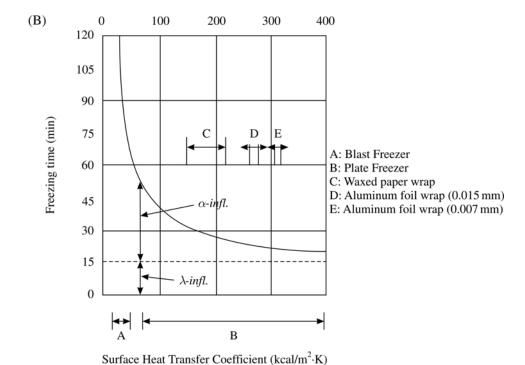
Figure 1B (120) shows the freezing time for 450 g fish fillet packs as a function of the overall heat transfer, including the influence of some types of packages used in contact freezing. This graph indicates that the type of packaging alone may increase the freezing time in a plate freezer by 2.5 times, and the surface heat transfer resistance by some 4 times. The freezing of packaged foods takes longer in an air blast freezer than in a plate freezer under comparable conditions. In the freezing of packaged food, where the  $\lambda$  value is influenced by the air inside the package, the difference in freezing rate between plate and blast freezing diminishes as does the influence of packaging material.

## C. Freezing Rate

Freezing must always be fast enough to minimize the development of microbiological and enzymatic changes in the product. A freezing process that occupies a matter of days will, in most cases, lead to deterioration in the frozen foodstuffs.

In the past, the beneficial effect of very rapid freezing on the quality of frozen foods has been overestimated: within certain limits the rate of freezing does not materially affect the quality of most foods. This should not be interpreted to mean that the rate of freezing has no effect on the quality of frozen foods; most foods suffer from being frozen very slowly; a few foods demand ultrarapid freezing. Fish and poultry seem to be more vulnerable to very slow freezing than most other foods, and meat (beef, pork, lamb) rather less. Strawberries and beans have a better texture and water holding ability if frozen ultrarapidly, while fruits and vegetables with a higher starch content such as peas are not as sensitive to freezing speed.





**Figure 1** (A) Freezing time in hours as a function thickness D of a slab of fish fillets frozen by removal of heat from two opposite sides of the package, for two values of the surface heat transfer coefficient  $\alpha = 20$  and  $\alpha = 200 \, \text{kcal/m}^2 \,^{\circ}\text{C}$ . A temperature of  $-30 \,^{\circ}\text{C}$  is assumed for the cooling medium (air, cold plates), and the process is considered to be completed when a temperature at the thermal center of  $-15 \,^{\circ}\text{C}$  is reached. The total freezing time is divided into two parts, one due to the influence of the internal conduction resistance ( $\lambda$ -infl.), the other to the surface heat resistance, including package ( $\alpha$ -infl.). (B) Freezing time in minutes for a 32 mm thick 450 g package of fish fillets as a function of the surface heat transfer coefficient. The total time is divided according to the influence of conduction resistance ( $\lambda$ -infl.) and surface resistance, including package ( $\alpha$ -infl.). The normal range of blast freezers and plate freezers is indicated. The influence of some types of packages in plate freezing is also shown.

In commercial practice, mean freezing rates vary between 0.2 and 100 cm/h; 0.2 cm/h (slow freezing) for bulk freezing in blast rooms, 0.5 to 3 cm/h (quick freezing) for retail packages in air blast or plate freezers, 5 to 10 cm/h (rapid freezing) for individual quick freezing of small sized products, e.g., in a fluidized bed, and 10 to 100 cm/h (ultrarapid freezing) by spraying with or immersion in liquid gases. For freezing of retail packages, freezing rates faster than 0.5 cm/h and for I.Q.F. products rates faster than 5 cm/h are considered satisfactory in most cases. Only very susceptible foods (such as tomatoes) may be improved by increasing the freezing to above 10 cm/h. At these rates care should be taken to avoid cracking. When freezing larger units, such as beef quarters, with a mean freezing rate of 0.1 cm/h, a freezing time of up to 5 days is unavoidable and times up to three days are quite commonly used.

The rate at which freezing takes place can be considered both at micro and at macro levels. At the micro level, freezing rate is described in terms of the speed with which the freezing front moves through the freezing object. At the macro level, the rate at which any given part of the object is cooled determines the temperature profile for that part and thus has an important bearing on the biochemistry and microbiology of that part.

The undesirable changes in meat during freezing are associated with formation of large ice crystals in extracellular locations, mechanical damage by the ice crystals to cellular structures through distortion and volume changes, and chemical damage arising from changes in concentrations of solutes. The fastest freezing rates are associated with the least damage (121). Differences in freezing rate modify meat properties. Ice crystallization and its growth in meat tissues are discussed by Calvello (122).

Freezing commences when the surface temperature of the meat reaches its freezing temperature. A continuous freezing front forms and proceeds from the exterior to the interior. Extracellular water freezes more readily than intracellular water because of its lower ionic and solute concentration. Slower freezing favors the formation of pure ice crystals and increases the concentration of solutes in unfrozen solutions. Intracellular solutions are often deficient in the nucleation sites necessary to form small ice crystals. Such conditions favor the gradual movement of water out of the muscle cells, resulting in a collection of large extracellular ice crystals and a concentration of intracellular solutes. Freezing damage arises from massive distortion and damage to cell membranes. Such effects have implications during thawing as the large extracellular ice crystals produce drip during thawing. The structural changes that occur also obliterate the recognizable muscle structure.

Fast freezing results in small ice crystal formation in both intracellular and extracellular compartments of the muscle and very little translocation of water. Drip loss during thawing is thus considerably reduced, and the surface reflects more light than that of slowly frozen meat. Consequently, the cut surface appearance is more acceptable.

## D. Freezing Methods and Equipment

Freezing equipment may be divided into the following main groups with regard to the medium of heat transfer. These groups are metal: plate freezers; air (gaseous medium) blast freezers; liquid: immersion freezers; and evaporating liquid: liquid nitrogen and liquid fluorocarbon equipment.

While blast freezers are used for all kinds of products packed or unpacked, blocks or I.Q.F. products, the plate freezer and the immersion freezer accept only packaged product, and evaporating liquid freezers are used only for I.Q.F. products.

#### 1. Plate Freezers

In a plate freezer the product is pressed by a hydraulic ram between metal plates which have channels for the refrigerant. This arrangement gives very good heat transfer of metal contact. This high thermal efficiency is reflected in short freezing times, provided the product itself is a good heat conductor, as is the case with fish fillets or chopped spinach. It is important that the packets be well filled and that the metal trays that are used to carry the packets are not distorted.

The advantage of good heat transfer at the surface is gradually reduced with increasing thickness of the product. For this reason the thickness is often limited to a maximum of 50 mm.

The pressure from the plates maintained throughout the freezing process practically eliminates the bulging that may occur in air blast tunnels; the frozen packets will maintain their rectangular shape within close tolerances.

There are two main types of plate freezers, horizontal plate freezers and vertical plate freezers.

a. Horizontal Plate Freezer. Usually this type has 15–20 plates (Fig. 2). The product is placed on metal trays, which are pushed in between the plates manually. This calls for a high labor content in the loading and unloading operation.

In order to obtain automatic operation of a horizontal plate freezer, the whole battery of plates is movable up and down in an elevator system. At the level of a loading conveyor, the plates are separated. Packages that have been accumulated on the conveyor are pushed in between these plates simultaneously discharging a row of frozen packages at the opposite end of the plates. This cycle is repeated until all frozen packages have been replaced. Then the space between the plates is closed and all plates are indexed up.

b. Vertical Plate Freezer. The vertical plate freezer has been developed mainly for freezing fish at sea. It consists of a number of vertical freezing plates forming partitions in a container with an open top. The product is simply fed from the top. The frozen block is discharged to the side, upwards, or down through the bottom. Usually this operation is mechanized, the discharge of product often being assisted by a short hot gas defrost period at the end of the freezing cycle and the use of compressed air to force the product out.

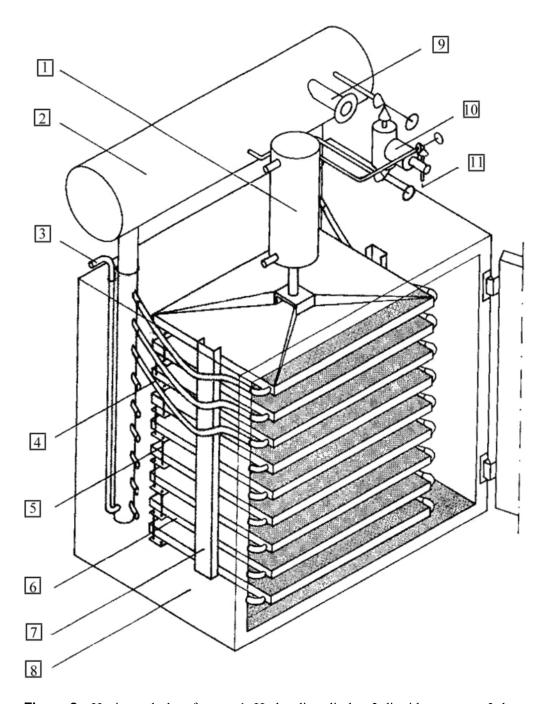
#### 2. Air Blast Freezers

Some foods, mainly bulk products such as beef quarters and fruits, for further processing are frozen in rooms with or without forced air circulation. Unless the room has been designed for freezing and equipped with suitable coolers and fans, the freezing rate is very slow, resulting in an inferior quality for practically all products. If the room is also used for storage of frozen products, the temperature of these products may rise considerably and the evaporators may frost up so quickly that the total refrigeration capacity is reduced below what is required to maintain the temperature of the store.

Good commercial practice for freezing in air blast uses include tunnel freezers, belt freezers, and fluidized bed freezers.

a. Tunnel Freezers. In tunnel freezers the product is placed on trays, which stand in or pass through the tunnel in racks or trolleys one behind the other or singly. An air space is left between the trays.

The racks or trolleys are moved in and out of the freezer by manual power or by a forklift truck (stationary tunnels), are pushed through the tunnel with a pushing mechanism (push-through tunnel), or are carried through by driving equipment, chain

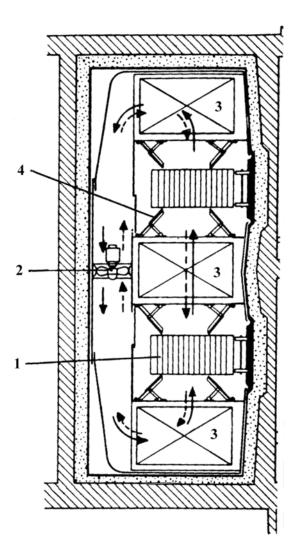


**Figure 2** Horizontal plate freezer. 1. Hydraulic cylinder; 2. liquid separator; 3. hot gas defrost; 4. flexible hoses; 5. link bolts; 6. freezing plate; 7. guide; 8. insulated cabinet; 9. suction outlet; 10. float valve; 11. liquid inlet.

drive, etc. (carrier freezer), or slid through (sliding tray freezer). Tunnels are also used for freezing hanging meat carcasses mostly carried on a suspension conveyor.

Tunnel freezers are equipped with refrigeration coils and fans that circulate the air over the product in a controlled way (see Fig. 3). Guide devices, properly locating the trays of food, lead to uniform freezing.

Tunnel freezers are very flexible freezers. Products of every size and shape, packaged or unpacked, can be frozen in stationary and push-through tunnels. Primarily they are used for freezing packaged products. Unpacked products tend to stick to the trays, which

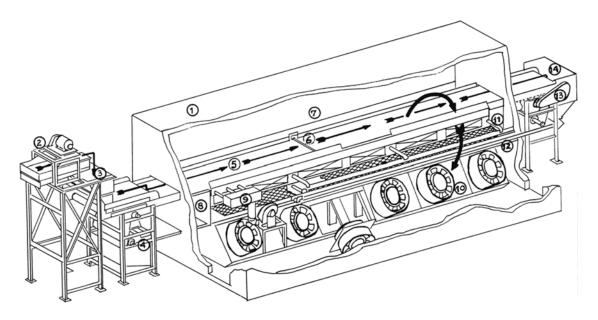


**Figure 3** Sectional drawing of a push-through tunnel freezer. 1. Trolleys with trays; 2. reversing fans; 3. air coolers; 4. system of baffles that allows adjustment of the airflow.

may cause weight losses and time-consuming handling in releasing, cleaning, and transport of the trays. To obtain free flow products, improved handling, and an increase in the freezing rate, individual quick freezing (I.Q.F.) is preferred.

b. Belt Freezers. Belt freezers are provided with a single belt (single-belt freezers, see Fig. 4) or to increase throughputs and to reduce floor space with belts positioned above each other that may run in the same or in opposite directions (multi-belt-freezers) or as a spiral belt wound round a rotation drum stacking up to 30 tiers of the belt above each other (spiral-belt freezers). The belt generally made of wire mesh remains inside the freezer so that ancillary equipment for in-and-out feeding is necessary. Alternatively, the belt is carried to the outside as in some spiral-belt freezers. This arrangement has the advantage that the products can be placed on the belt in the processing room, where the operation can be supervised, before entering the freezer, and the product will remain undisturbed until removed at the outlet (Fig. 4). The belt is supported by rails and driven by passing round the rotating drum.

Modern belt freezers have vertical air flow so that the air is forced through the product layer. In freezing small products such as beans or cherries, good contact with all



**Figure 4** Single belt freezer. 1. Insulated wall of the tunnel; 2. de-watering vibrator; 3. loading hopper; 4. belt drying system; 5. variable speed belt (open mesh belt); 6. product spreader; 7. air agitation zone; 8. evaporator; 9. high-velocity air; 10. variable air flow fans; 11. defrost water; 12. refrigerant piping; 13. belt speed changer; 14. unloading hopper.

product particles is thus created. In single-belt freezers with high air velocities the products may agitate. In all belt freezers care should be taken to spread the product uniformly across the total belt width to avoid "channeling," where the air stream bypasses the product.

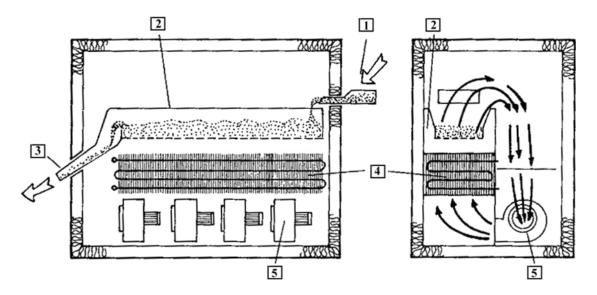
Belt freezers are used mainly for freezing unpackaged products, e.g., I.Q.F. products. They are specially suitable for foods that need careful handling.

c. Fluidized Bed Freezer. Fluidization occurs when particles of fairly uniform shape and size are subjected to an upward air stream. At an air velocity depending on the characteristics of the product, the particles will float in the air stream, each one separated from the others but surrounded by air and free to move. In this state, the mass of particles behaves like a fluid. If the product is contained in an inclined trough that is fed at the higher end, the fluidized mass moves toward the lower end, as long as more product is added. The product is thus frozen and simultaneously conveyed by air without the aid of a mechanical conveyor (Fig. 5).

The use of the fluidization principle has the following advantages when compared with a belt freezer:

- 1. The product is always truly individually frozen (I.Q.F.). This applies even to products with a tendency to stick together, e.g., French style (sliced) green beans, sliced carrots, and sliced cucumber.
- 2. Independence of fluctuations in load. If partly loaded, the air distribution will be the same as for a full load, i.e., no hazard of channeling. If over-loaded, no product flows onto the floor.
- 3. Reliability is improved when freezing wet products, because the deep fluidized bed can accept products with more surplus water.

An important factor in the overall operation economy of a blast freezer is the weight losses during freezing. Improperly designed equipment will have losses of 5% or more



**Figure 5** Fluidized bed freezer. 1. Unfrozen product conveyor; 2. product trough; 3. frozen product discharge; 4. air coolers; 5. fan.

while a well designed freezer normally operates with only 0.5 to 1.5% loss for unpackaged products. Part of the weight losses are dehydration losses, which require particular consideration. Weight loss is minimized by low air temperatures and good heat transfer, i.e., high air velocities.

A freezing tunnel that is intended for packaged products should not, without due consideration, be used for thin unpackaged products, e.g., fillets of fish. The result may be that the relation of coil surface to product surface is put out of balance so that air temperature in the tunnel rises with resulting high weight losses. The coil may not be able to accommodate sufficient quantities of frost, which results in reduced heat transfer or reduced air flow, both contributing to high weight losses.

It is important to note that in a vapor tight package containing a product that is not homogeneous, e.g., beans or broccoli, the heat transfer inside the package is carried out by air. The heat transfer is very poor, because there is no air circulation. The result is evaporation of moisture, which actually may be greater than it would have been without the package. This moisture remains as frost on the inside surfaces of the package, so that it is not usually recorded as a weight loss. The influence on product quality is, of course, the same whether the dehydration is recorded or not.

Higher velocities of air give better heat transfer. However, it is not sufficient just to increase the fan power. The most important factor is to direct the air circulation in such a way that every product particle is efficiently and equally exposed to the air current. It is also important to study the conditions of the individual particles, because a close study may reveal surprising uneven air flows.

#### 3. Immersion Freezer

For irregularly shaped products, e.g., chicken, the best heat transfer is achieved in an immersion freezer. This consists of a tank with a cooled freezing medium, e.g., a salt or propylene glycol solution. The product is immersed in this brine or sprayed while being conveyed through the tank.

Immersion freezers are most commonly used for surface freezing of poultry to obtain a good color. The final freezing is affected in a separate blast tunnel or cold store. The latter alternative, however, involves quality hazards because of the slow freezing of the core.

The product must be protected by an extremely tight, high quality packaging material. The brine on the package is washed off with water at the exit of the freezer.

#### 4. Evaporating Liquid Freezers

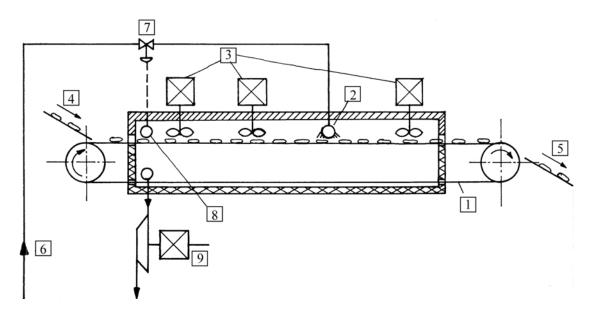
Mainly two liquids or freezants are used, liquid nitrogen (LN2) and liquid fluorocarbon freezant (LFF).

a. LN2 Freezer. Liquid nitrogen at  $-196^{\circ}$ C is sprayed onto a single-belt freezer. The nitrogen evaporates and is allowed to escape to the atmosphere after the vapors have been used for precooling of the products (Fig. 6).

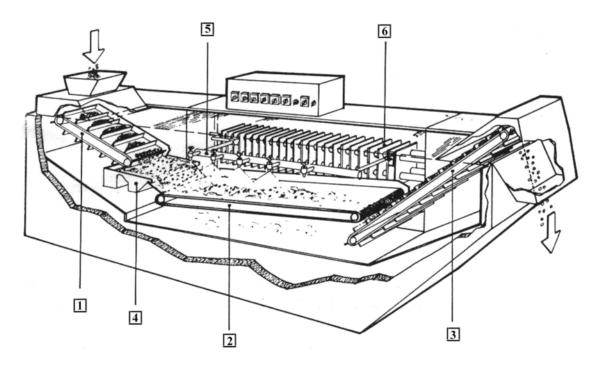
The very high freezing rate results in improved textures, particularly in certain fruits and vegetables, while with other products there seems to be little quality advantage compared with other freezing methods. LN2-freezing may result in cracking of the product surface if sufficient precautions are not taken.

Like immersion freezers, LN2 freezers are often used only for surface freezing. If final freezing is to be carried out, the LN2 consumption is of the order of 1.0–1.5 kg per kg of product, which makes the operation rather expensive. In spite of this, the low investment and simple operation make this method economical for certain productions, especially in-line processes.

b. LFF system. The freezant is a specially purified dichlorodifluoromethane (fluorocarbon), which has a boiling point of  $-30^{\circ}$ C at atmospheric pressure. The equipment consists of a container with openings at the top. The product is introduced into the container and dropped into a flowing stream of freezant (Fig. 7). Owing to the extremely good heat transfer, the surface is frozen instantaneously so the product may be stacked on the horizontal freezing belt, where it is sprayed with freezant until finally



**Figure 6** Liquid nitrogen freezer. 1. Belt; 2. spraying nozzles; 3. fans; 4. inlet; 5. outlet; 6. nitrogen tank supply line; 7. regulating valve; 8. temperature sensing unit; 9. nitrogen gas exhauster.



**Figure 7** Liquid fluorocarbon freezer. 1. Product input conveyor 2. freezing conveyor; 3. frozen product exit conveyor; 4. I.Q.F. bath; 5. spray nozzles; 6. condenser.

frozen. A discharge conveyor brings the product up and out of the freezer. It is claimed that fluorocarbon leaves only small residues in most products. Experiments in this area are continuing.

On contact with product, the freezant evaporates. The vapors are recovered (with only a slight loss) by condensation on the surfaces of the refrigerator, the latter remaining in the container with only small losses to the atmosphere. There is no measurable product weight loss due to dehydration using this method.

#### XI. STORAGE

If the quality of frozen food is to be maintained during its storage life, the correct temperature must be selected for the expected period of storage. During the storage period, the following hazards to quality must be avoided:

- 1. A low relative humidity in the cold store
- 2. Retention beyond the expected storage life
- 3. Fluctuations in temperature (both during storage and in the process of loading, unloading, and dispatching vehicles)
- 4. Physical damage to the product or packaging during the course of storage or handling
- 5. Contamination of the product by foreign bodies or vermin

These hazards can be avoided by ensuring

1. That the design of the cold store is appropriate to the duty it will be required to perform and is such that these hazards are as far as possible eliminated at the design stage.

2. That operation methods, designed to avoid such dangers, are laid down and strictly adhered to.

Small fluctuations in temperature are normal and unavoidable. They should, however, be kept at a minimum both in amplitude and in duration in order to minimize the amount of weight loss by drying and in-package desiccation.

## A. Cold Store Design

The general design of a cold store is determined by the requirements for effective and safe handling of the merchandise, and a suitable storage climate for the products. The normal arrangement is that rooms are built side by side between road and railway loading banks, so that all rooms can communicate directly with the loading banks and traffic yards. Today cold stores are frequently built with prefabricated concrete or steel structures. The insulation can be placed on the outside or the inside of the structure. Internal structure means that the insulation will form an unbroken envelope around the building. Thus the insulation is well protected by the structure, internal installations are easy to fix, there is no hung insulated ceiling that can cause problems, and extensions are very simple to carry out.

It is essential that considerable thought should go into the definition of the duties the cold store has to perform. A clear statement has to be prepared of the maximum and average daily activity expected to take place in the cold store:

- 1. Quantity of each product to be received
- 2. Temperature at which each product will be received
- 3. Maximum number of operatives and trucks working in the cold store at any one time
- 4. Number of anticipated door openings
- 5. Maximum quantity to be outloaded at any one time

These considerations must be taken into account when calculating the maximum expected heat load. The temperature difference between the surface area of the cooling coils and the required room temperature should be as small as possible and not more than about  $6^{\circ}$ C.

#### 1. Frost Heave

Frost heave under cold stores is prevented by a special under-floor heating system or a ventilated space under the floor. The heating system may consist of an electrical mat or a pipe grid cast into the subfloor; glycol or oil is circulated in the pipes. The liquid is often heated by surplus heat from the refrigeration plant.

#### 2. Insulation

The insulation represents a large percentage of the total cost for a normal cold store. It is therefore very important that it be designed from an economical point of view. However, one must also consider that the insulation value has an influence on the storage climate in that the transmission losses mean that dry heat is entering the cold room. The choice of insulation system must be carried out carefully. The vapor barrier on the warm side must be completely water-vapor-tight, the insulation should contain no heat bridges, and the internal cladding must be hard, hygienic, and of pleasant appearance. Today most cold

store insulations are carried out with prefabricated panels, slabs of polyurethane foam, expanded polystyrene, mineral fiber, or cork. Where special attention must be given to the risk of fire, the insulation is combined with a special fire wall or the insulation is carried out with fiberglass between special insulation studs. The vapor barriers may consist of thin-gage aluminum, galvanized steel sheets, or heavy-gage polyethylene sheets. The joints are sealed with special sealing compounds. The internal cladding may be profiled plastic, laminated galvanized steel sheet, or aluminum sheet. Good concrete kerbing and in some cases dunnage battens protect the internal finish and ensure that the merchandise is not stacked directly against the wall.

#### 3. Refrigeration System

The refrigeration system must be designed with regard to the requirements of the climatic conditions for the stored merchandise. It must be adequate to allow for sufficient safety on peak days and summer conditions. The air coolers must be designed and located so that an even temperature can be maintained throughout the cold store even under severe conditions and without generating high air velocities in the cold store. Large evaporator surfaces and air distribution through air ducts or false ceilings will normally ensure this. Air ducts may be omitted if the cooling surface is divided on several cooler units distributed in the room so that the air velocity from the cooler fans is kept at a moderate level.

The most common refrigeration system for large cold stores is a two-stage compressor system with pump circulation of the cold refrigerant to the air coolers. The most common refrigerant is ammonia, but halogenated hydrocarbons have also been used in some cases. For small cold stores, a direct-expansion one-stage compressor system using halogenated hydrocarbons is widely used. In order to improve safety and make control easier and cheaper, most modern refrigeration plants are automated. The degree of automation may vary, but normally the room temperature, compressor capacity, lubrication, cooling water, defrosting, pumps, fans, and current and voltage of the main supplies, etc., are controlled and supervised by a central control panel in the engine room.

#### 4. Lighting

A cold store is a working place for forklift drivers and others concerned with the handling of the products. Thus the lighting in the cold store must be good, but at the same time it must be remembered that the lighting is adding to the heat load in a cold store. Lamps with a very high power/lighting ratio should be used. Mercury lamps are superior from this point of view, and they are often used even if they can cause a slight discoloring of meat products during long storage. A normal cold store should have an average lighting of 100 lux at floor level and in breakup areas 200 lux.

#### 5. Layout

The layout of the storage space should be such as to reduce to the maximum extent possible the ingress of warm air and the exposure of product to atmospheric temperatures. Where possible, product should be conveyed from factory areas into cold store by means of conveyors in insulated tunnels. There should be no facility for any accumulation of product in ambient temperature. If the operation is a palletized one, then palletization of the product should take place in the cold store in an area set aside for this purpose. Port doors should be provided so that the maximum amount of traffic in and out is handled in

this fashion and the product is completely protected from temperature changes during loading/unloading operations.

For safety, no glass should be allowed inside the cold store in any unprotected position. Translucent plastic visors should be placed around lamps or any other essential glass.

#### 6. Jacketed Stores

Jacketed stores allow storage at near 100% relative humidity and at uniform and constant temperatures. These conditions, which greatly reduce weight losses of unpackaged foods and frost formation inside packaged frozen foods, are obtained by circulating the refrigerated air in a jacket around the load space to absorb the heat conducted through the insulation before it can enter the load space (Fig. 8). This technique also increases the life and reduces the maintenance of the structure by preventing condensation and frost formation in the insulation.

## 7. Equipment

In equipping the store, care should be taken to choose equipment that is suitable for the product being handled and that minimizes the possibility of damage or contamination; thus timber pallets are suitable for properly packed products, but lightly packed semiprocessed stock may need a pallet constructed of metal or some similar washable and less easily damaged material.

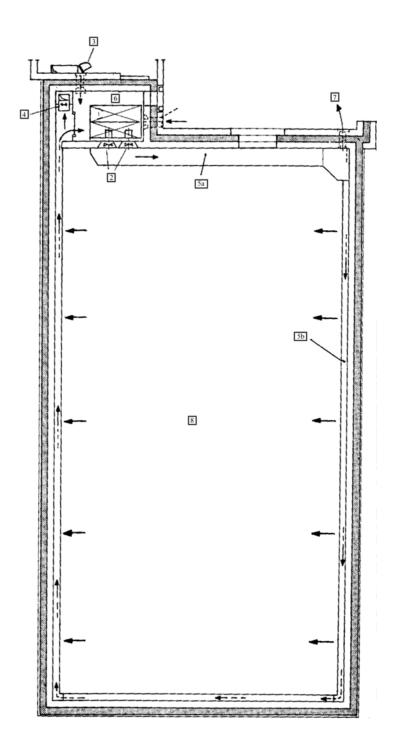
It is likely, for economy reasons, that the store will be designed to maximize the use of height, and to this end some means of support for the product must be provided; e.g., racks, pallet posts, or pallet cages. The layout of pallets in the cold store should be such that damage to the products is minimized. Whilst accepting the need for maximum utilization of space, gangways and turnings should be wide enough for product to be moved without damage, while space should be allowed between lanes of pallets to permit the withdrawal or placing of a pallet when the adjacent lanes are full.

## 8. Operating Methods

Everyone working in a cold store should always bear in mind the prime objective of minimizing the exposure of products to ambient temperatures. Methods of handling and routes should be laid down that do not permit the product being placed in ambient temperature. If it is not possible to load a vehicle by means of a port door or some similar method that gives complete protection, and the only alternative is to traverse a loading bank, then the complete vehicle load should be assembled inside the cold store and conveyed direct to the vehicle without being placed on the loading bank. A similar procedure should apply in reverse for unloading. Doors should never be left open other than when personnel or goods are passing through them, and these should be as short a duration as practicable.

#### XII. THAWING

Most frozen food processors find it necessary to thaw stock of frozen material in some of their operations, and if thawing is not carried out carefully, quality and yield can suffer. This section sets out the broad principles involved in the thawing techniques available and indicates some of the problem areas. Irrespective of the procedure involved, heat energy



**Figure 8** Jacketed store. 1. Air coolers; 2. fans; 3. fresh air duct; 4. air vent; 5a. air ducts; 5b. jacket; 6. air cooler enclosure; 7. cooling orifice to antechamber; 8. cold room. This type of jacketed store serves two purposes, thanks to the two-speed fans. At the lower speed, the air only circulates in the jacket. This is the classic operation of jacketed stores (arrows marked  $--\rightarrow$ ). At the higher speed, the air enters the store through a system of movable vents that open via the effect of the increased air pressure. Through this means, one can complete the cooling of foodstuffs that may have been subjected to warming prior to being introduced into the cold store (arrows marked thus  $\rightarrow$ ).

must be supplied, most of it being required to melt the ice in the food. About  $300 \, \text{kJ}$  are required to thaw 1 kg of white fish from a starting temperature of  $-30^{\circ}\text{C}$ . The figure for fatty herrings is smaller, about  $250 \, \text{kJ}$ , because of the lower water content of herring. Thawed materials spoil in the same way as their unfrozen equivalents and must be kept chilled until required; this can often be achieved conveniently by removing the product from the defroster just before thawing is complete so that the product has its own small reserve of "cold."

There are two principal groups of thawing methods: those in which heat is conducted into the product from the surface; and those in which heat is generated within the product. In the first group, heat is applied to the surface of the product by exposing it to sources such as hot radiating surfaces, warm air, warm water, heated metal plates, or steam under vacuum. In the second group, heat is generated within the product by such means as electrical resistance and dielectric or microwave heating. Surface heating methods are much more commonly used than are internal heating methods.

# A. Surface Heating Methods

When using surface heating methods, thawing time for a product decreases with

- 1. Decrease in physical size of the product
- 2. Increase in thermal conductivity (or, more precisely, thermal diffusivity) of the thawed product
- 3. Increase in temperature difference between the surface of the product and its surroundings
- 4. Increase in the movement of the surrounding medium relative to the product surface
- 5. Increase in humidity of the surroundings

Since the thermal diffusivity of thawed product is less than that of frozen, surface thawing methods suffer from the inherent disadvantage that resistance to heat transfer increases progressively once thawing has started.

All surface heating methods can take advantage of a programmed temperature difference between the surface and the surroundings; the temperature of the surroundings is arranged to start high, and to decrease as the surface warms up to a predetermined level, usually below the temperature where bacterial activity or surface damage could be a hazard to the quality of the food. Thawing times are greatly increased if the product is encased in packaging material. Thus packaging material should be removed where possible.

# B. Still Air Thawing

If thawing is conducted in still air, the air temperatures should not exceed 20°C, and facilities for supplying heat to the room in which the product is laid out to thaw may be required. Air temperatures greatly in excess of the above should be avoided, since the outer layer will warm up and spoil before the center is completely thawed. A typical block of sea frozen whole cod 9 cm thick will take up to 20 hours to thaw under these conditions. This time can be reduced by separating the fish as soon as these can be separated without damage. Single fish, 10 cm thick, will take about 8 to 10 hrs to thaw.

# C. Air Blast Thawing

A relative humidity of greater than 90% is advantageous both in reducing weight loss or shrinkage and also in obtaining a high level of heat transfer. Air velocities of 12–18 m/min at a temperature of 6–8°C for 3–5 days or 100 m/min for 2–2.5 days are needed for packages of boneless meat, whereas pork sides are normally thawed at 4–5°C for 2–4 days. Air speeds of the order of about 300 meters per minute at temperatures not exceeding 20°C with the air fully saturated with moisture will thaw sea frozen fish blocks 10 cm thick in 4 h. Higher air velocities at cool temperatures lead to surface desiccation, while any higher temperature will lead to microbial growth on the heated surface.

## D. Water Thawing

This method is not normally applicable to meat, and with fish there is a risk that fillets or cut surfaces will become waterlogged and lose flavor. But water thawing can be used satisfactorily for frozen whole fish, even though there may be a slight loss of pigments. Usually there is a slight gain in weight, which is lost again when the fish is filleted.

The temperature of the water being circulated around the frozen fish or sprayed onto the fish should be no warmer than 20°C, and the water itself should flow at not less than 30 cm per minute to obtain rapid thawing. The thawing time for a block of whole cod 10 cm thick in water at 20°C moving at about 120 cm per minute is similar to that in an air blast defroster using humid air at the same temperature, i.e., about 4h.

When frozen fish blocks are thawed in still water, water temperatures may, in the initial stages, marginally exceed  $20^{\circ}$ C, but the thawing should be arranged so that the fish surface temperature does not exceed  $20^{\circ}$ C.

### E. Vacuum Thawing

In this method the product lies on racks inside a container from which the air has been evacuated. Water, usually at about 18°C, is allowed to evaporate freely from heated vessels inside the container. In the absence of air, transfer of water vapor from a heated vessel to the product occurs readily, the water vapor giving up its latent heat on condensation. The advocates of this method claim faster thawing than other surface heating methods for products less than about 10 cm thick.

### F. Double Contact Thawing

Plate frozen raw material lends itself particularly to plate thawing in an arrangement similar to that of a multiplate freezer, a liquid circulating through the plates (at a temperature not exceeding 20°C) providing the necessary heat. A 10 cm block of whole cod when thawed between plates at 20°C for 5 h is ready for filleting 3.5 h later, making a total of 8.5 h for complete thawing. Care must be taken not to allow distortion of the blocks to occur during cold storage, since this will lead to poor contact with the plates during thawing. Thawing fluid or semifluid food in a vertical plate apparatus may be carried out using temperatures as high as 40–50°C as long as the melted material is allowed to run away from the plates continuously.

# G. Internal Heating Methods

Internal heating methods rely on the use of applied electric fields that cause movement of the electric charges inherent in all products. The molecules of the product take up this energy of movement and the food warms up. The amount of heat generated in this way is strongly dependent on the electrical characteristics of the product. Since food is not usually homogeneous, there can be marked variations in the rate of heating of different parts of the food. Furthermore, for any particular component in the food, the rate of heating usually increases as the product thaws, making runaway heating a hazard. If these factors can be accommodated, the great advantage to be gained is extremely rapid, uniform thawing.

# H. Electrical Resistance Thawing

In this method, the product is sandwiched between electrodes and an electric current is passed through the product. Some preliminary warming is usually necessary to achieve good electrical contact and a satisfactorily high starting current. In practice this method has so far been used for thawing blocks of fish fillets up to 5 cm thick and weighing about 3 kg, but it has not been found suitable for thicker blocks of fillets, blocks of whole fish (except herring), or single whole fish.

# I. High-Frequency Thawing

In the dielectric method, a high-frequency electric field is applied to electrodes astraddle the product, but not physically in contact with it. The commonly used frequencies are either in the range 27 to 100 MHz (dielectric or high frequency heating) or 915 to 2450 MHz (microwave heating; where the energy is directed at the product enclosed in a chamber). The product must be reasonably homogeneous and regular in shape to achieve uniform heating. If the block is not homogeneous, or is irregular in shape, some parts may become overheated before the remainder is thawed. It has been found that if irregular blocks of fish are first immersed in water, thawing becomes uniform. The time taken for a 10 cm block of whole cod is typically just less than 1 h. Partial thawing by microwave is also used, thereby increasing the capacity of the thawing equipment. By going to the higher frequencies, the field strength can be substantially increased and thawing time reduced to a matter of minutes. Penetration into the food mass decreases so that the thickest block of meat that can be completely thawed at 2450 MHz is 3 to 4 cm.

### XIII. CONCLUSIONS

In summary, freezing, frozen storage, and thawing affect the quality and shelf stability of fish and seafood. If kept under appropriate conditions, fish and seafood can be stored in the frozen state for several months without appreciable changes in quality. During frozen storage, microbiological changes in fish and seafood are minimal. On the other hand, a series of changes such as protein denaturation, lipid oxidation, texture deterioration, loss of fresh odor and flavor, various enzymatically induced reactions, loss of volatile constituents, nutritional losses, and changes in moisture take place in fish and seafood when subjected to excessively prolonged frozen storage. Likewise, such changes may also occur in freeze—thawed fish and seafood. Quantitative evaluation of the influence of

freezing, frozen storage, and thawing on fish and seafood is rather complex. The different variables that influence quality are related to one another. Therefore it becomes almost impossible to describe some quality changes without actually discussing the other related changes that occur in fish tissues.

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# **17**Freezing Finfish

### B. Jamilah

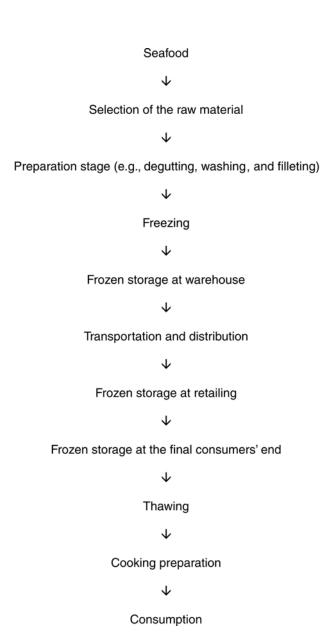
Universiti Putra Malaysia, Selangor, Malaysia

### I. INTRODUCTION

Seafood is available fresh, chilled, and frozen as unprocessed, minimally processed, or processed as, for example, fabricated and breaded products. Freezing fish and fishery products makes the commodity available throughout the year and also in regions where they are not produced. Advancement in freezing technology gives frozen foods eating qualities comparable to those of freshly prepared foods, but knowledge of the character of the commodity and of the handling requirements throughout the cold chain still plays a big role in ensuring the maximum attainable quality attributes. Figure 1 shows the stages of handling of the seafood in its life as a frozen food commodity. For fish and fishery products, handling at the stage of preparation, understanding chemical changes such as lipid oxidation and protein denaturation, textural changes, and changes in the overall quality during frozen storage are crucial. The mishandling of frozen products can occur anywhere along the cold chain, for example, at retail and at the consumer's end. The quality maintenance of the frozen seafood could be segmented into (a) prefreezing selection and preparation, (b) during freezing, and (c) postfreezing handling—frozen storage at warehouse, retailing, and the consumer's end, and (d) thawing. Fish tissues differ with species, composition, catch, and freshness (1–5). These differences cause the freezing characteristics of the fish to differ. Quality deterioration in frozen seafood exhibits different phenomena than those observed in chilled or ambient storage.

### II. PRINCIPLES AND APPLICATION IN FREEZING SEAFOOD

The general guidelines for good manufacturing practice for freezing food commodities are also applicable for the freezing of seafood with the extra precaution on the need to minimize lipid oxidation and textural changes. These two are the main elements that contribute to the quality characteristics of the product besides the other specific product identity.



**Figure 1** The flowchart of the stages in the life of a frozen seafood product.

# A. Quality Attributes Before Freezing—Prefreezing Handling

This is the first step in the series of quality control measures for the production of frozen seafood. The steps at this stage vary with the product. To ensure the best quality, the factors here should be considered.

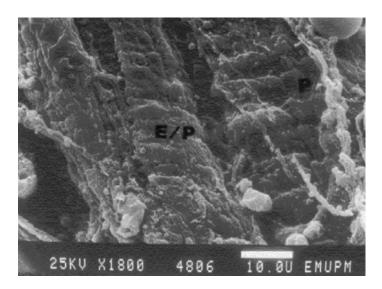
- 1. Selection of the raw material, which may include product characteristics such as the body composition, size, and suitable species
- 2. Freshness of material
- 3. Product uniformity for processed products such as nuggets, balls, and breaded products
- 4. Presence of a quality control and assurance system to enable fast and effective remedial action when needed

5. Packaging design and selection of the packaging materials, the bulk weight of the individual package, and the recommended serving size

Characteristics and Handling of Raw Material. Pioneer works in the freshness of fish have been credited to Japanese scientists. Fish freshness deteriorates very rapidly. This rapid deterioration is due to the naturally high enzymatic activities in the fish muscle as compared to those in other food commodities. The natural sweetness of fish muscle is related to its degree of freshness. High ambient temperatures, temperature fluctuations during storage, delay in icing or chilling of unprocessed material, and improper thawing practices of frozen raw material such as frozen whole fish will expedite the rate of deterioration. The rate of deterioration is governed by the species and the origin of a catch, as demonstrated by Johnston et al. (6) in their comparative study of 19 species of fish. They reported that myosins of warm-water species are more stable at the studied temperature (0–18°C) than fish from temperate waters. This can indicate that delay in lowering the temperature in handling temperate fish will result in faster spoilage. In 1986, the speed of lowering in freshness for 82 species of fish comprising species from tropical, subtropical and temperate waters was reported (7). Their results pointed out that the rate of freshness loss was generally slower for warmwater fish. Preparation steps for freezing, for example, washing and glazing, will not eliminate the degradative processes already taken place, such as changes in chemical composition and organoleptic properties. As a guideline to ensure that only good quality material is selected as the raw material, the factors listed here should be considerd:

- 1. Only fresh fish should be frozen or processed for the final frozen products.
- 2. Physically damaged fish should be discarded.
- 3. Adequate cooling of fish and fishery products prior to freezing.
- 4. Adequate protective measures to prevent surface dehydration.
- 5. Ensure product uniformity.
- 6. Avoid filleting during rigor.

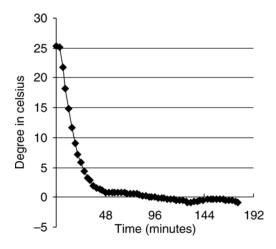
Chilling of fish with ice or a mixture of ice and water is a common practice during handling in the majority of landing sites to delay the onset of spoilage. Walk-in chillers are often used in processing premises for cooling of raw materials prior to processing. If left for an extended period, surface dehydration of the upper layer of fish and softening of the muscle can take place owing to the autolytic process. Prerigor muscle is always the best starting material for further processing. During rigor, the muscle goes through a natural contraction process, which is a resemblance of a tensed-up muscle. The onset of rigor and its duration vary with species and its history of handling. Figure 2 of the microstructure of the bighead carp muscle in the state of rigor (after 8 h at ambient temperature) clearly shows crinkling as the effect of muscle contraction. A similar microstructural description for blue grenadier (Macruronus novaezelandiae) was also reported (8). Structural changes of the fish flesh and related biochemical changes such as protease activities postmortem has been well discussed by Bremner (9). Gaping, the visual separation of the myotome, the fish muscle block, has been associated with filleting during this stage for cod (10). Owing to the concurrent lowering of the water-holding capacity of the fish muscle in the rigor stage, the filleting process is not generally recommended. Freezing during rigor is also not generally recommended, since frozen storage may arrest the normal process of muscle relaxation, i.e., resolving of rigor.



**Figure 2** The scanning electron micrograph of bighead carp (Aristichthys nobilis) muscle in rigor (P = perimysium; E/P = endomysium/plasmalemma).

# B. Quality Attributes During the Freezing Process

The freezing process that a seafood product undergoes is similar to that of other food commodities. A comprehensive review on the mathematical approach of predicting freezing rates of food have been documented (11). Differences may exist in the actual freezing point and the superchilling phenomenon. The freezing point and the superchilling also differ in seafood products. Figure 3 shows part of a typical freezing curve of Indian mackerel (*Rastrellinger kanagurta*) in the round caught in Malaysian waters in the year 1985 as obtained during plate freezing set at  $-20^{\circ}$ C. The superchilling characteristic could barely be observed in the vicinity of  $-0.8^{\circ}$ C (the experimental freezing point was approximately  $-0.5^{\circ}$ C). The freezing technique employed depends on the products manufactured. Considerations such as unit size and the economics of the production usually influence the decision made. Leniger and Beverloo (12) suggested three groups of



**Figure 3** A typical freezing curve of Indian mackerel obtained in plate freezing.

freezing rates: fast freezing (>5 cm<sup>-1</sup> h), moderately fast (1 to 5 cm<sup>-1</sup> h) and the slow freezing ( $< 1 \,\mathrm{cm}^{-1} \,\mathrm{h}$ ). The difference in these freezing rates is said to be contributed by four factors: the temperature difference between the product and the cooling medium; the modes of heat transfer to, from, and within the product; the size, shape, and type of package containing the product; and the size, shape, and thermal properties of the product (13). A recent study conducted on the freezing rate of tilapia (Oreochromis sp.) muscle by air blast and liquid nitrogen freezing has indicated that the freezing rate of the muscle is strongly correlated to the freezing temperature, i.e., the lower the temperature, the faster is the freezing rate, regardless of the technique used (14). Fast freezing has always been advocated for a superior quality product. Cryogenic freezing has been recognized as a very rapid freezing technique and is gaining acceptance, minus the financial constraint. Freezecracking, a condition associated with crust formation, may happen in some cases; for example, freezing whole fish cryogenically has been reported to induce thermal stress and cause a radial cracking and a shattered structure upon thawing (15). Some reports, however, imply that fast freezing will ensure better storage stability of the fish during frozen storage (16–18).

The rate and size of ice crystal formation are dependent on the rate of freezing. Jul (19) gave a good review of the historical background on freezing rate studies conducted during the early era of the frozen food industry. Rapid freezing has always been associated with small ice crystals, thus causing minimal textural damage upon product thawing.

# C. Quality Attributes During Frozen Storage

Examples of some of the quality attributes of frozen seafood that are of concern are the general appearance, such as the presence of excessive ice crystals in the package and on the product surface, surface discoloration, and integrity of the package. Ice formation in the package and on the product surface is due to moisture migration from inside the product to the outside environment owing to the temperature and the vapor pressure difference in the package (20, 21). Other quality attributes such as textural change and change of flavor and odor are only recognizable upon consumption. It is also important to have a careful monitoring of warehouse temperature, duration of storage, and logistics. Improper retail handling and display can cause quality deterioration. It has been observed that temperature fluctuation of the frozen product happens quite often during the defrosting of the display refrigerators. The extent of the quality deterioration depends on the number of freeze-thaw cycles experienced by the product. Loosening of breading material or loss of product original shape, for instance, in reformed products, can cause great loss. Issues concerning freshness and its relation to fish quality have been discussed and reported in numerous fish species. Loss of degree of freshness of unprocessed and minimally processed fish continues during frozen storage, although at a slower rate. Prolonged frozen storage for 52-54 weeks at  $-20^{\circ}$ C is more detrimental to fish species from a cold water environment than it is to those from a warm water environment, based on the enthalpies of the transitions of their myosin in a differential scanning calorimetry (DSC) study (22).

# D. Product Thawing

Frozen seafood is generally thawed prior to final cooking or heat treatment. Breaded products, however, are quite exceptional. Improper thawing can make the efforts of a good frozen storage condition fruitless. Thawing can be done in the chiller (1 to 5°C) overnight as practice both in the industry and in some households. Thawing in chillers may take slightly

longer when the bulk size of the product is greater. Microwave thawing is used in some fast-food chains. Thawing at room temperature or in water can also be carried out, and it is usually recommended not to exceed 2 h. The drop in the quality of frozen seafood upon thawing is related to the drip loss in the unprocessed or the minimally processed products. Excessive thawing will result in loosening of the breading material. Greater drip loss is associated with the formation of bigger ice crystals during slow freezing.

### III. SHELF LIFE OF FROZEN SEAFOOD

The shelf life of frozen seafood is shortened when the expected quality of the commodity falls short of the expectations of consumers. Slightly different variables affect the shelf life for unprocessed or minimally processed foods and those that have received some extended processing steps.

The shelf life of unprocessed seafood is usually affected by changes in the natural texture, flavor, and perhaps the surface color and appearance. The presence of additives, texture modifiers, flavor enhances, breading materials, processing steps such as mixing time, and heat treatment processes such as steaming, boiling, and frying are some of the additional factors that will eventually influence the shelf life of the processed product. The shelf life of reformed products such as those formulated with surimi as the main protein as compared to some formulated from minced fish instead will have different elements to dictate their respective shelf life. Freeze stability of surimi-based products are also dependent on the water binding agents added such as natural biopolymers and cryopectants (23). There are abundant studies on the mechanism of quality deterioration or factors affecting shelf life during a selected duration of frozen storage at a set temperature. However, articles on the actual shelf life, simply understood as the acceptable duration of palatability of the product, are not common. Sensory studies using panelists are required to supplement this information. An overview of using sensory evaluation to determine the shelf life of frozen foods is covered by Jul (19). Table 1 summarizes reported shelf life of some seafood products.

The shelf life of frozen fish is species dependent beside the techniques of freezing and the actual frozen temperature employed. The fatty acid composition, the proportion of red to white muscle, the specific odor development, the characteristic textural changes, and the history of prefreezing handling are some of the contributing factors to the shelf life of frozen fish. Handling abuse at the terminal of the cold chain as seen in Fig. 1 is detrimental to the quality of the product. Quality lowering is often associated with protein denaturation and lipid oxidation in one way or another.

Change in the texture of the frozen fish is displayed as the toughening of the muscle or described as the development of a fibrous texture. Textural changes in frozen fish muscle have been attributed to the changes in the water binding ability of the fish myofibril upon thawing. Differential scanning calorimetry (DSC) provided evidence that the myosin is affected during the process of freezing owing to the unfolding of the molecule and its ability to rehydrate upon thawing. This is also affected by the rate of freezing (24). An electrophoretic study on myosin denaturation of white and red muscles from cod (Gadus morhua), tusk (Brosme brosme), and capelin (Mallotus villosus) also pointed to the different sensitivities of the myosin molecule of different fish species and tissues (25). Excessive duration in the frozen state also exemplifies these textural changes. Pronounced textural changes of hake (Merluccius merluccius) frozen for 2 years at  $-12^{\circ}$ C were attributed to the aggregation of the myofibrillar protein and collagen (26). It has also been suggested that

**Table 1** The Shelf Life of Some Frozen Seafoods

Type of seafood	Approximate shelf life	Source (Ref.)	Remarks on quality deterioration
Breaded fish finger	6–12 months at −18 °C	36	Not available
Breaded tilapia fillets	$4-5$ months at $-20^{\circ}$ C	37	Development of fibrous texture
Breaded catfish (Clarius batrachus) fillets	6 months	38	Toughening of texture
Burger (prawn and fish)	6 months at $-18$ °C 1–3 months at $-10$ °C	36	Not available
Cuttle fish ball	6 months at $-20^{\circ}\text{C}$	36, 39	Toughening of texture
Fish sausage	6 months at $-20^{\circ}\text{C}$	36	Not available
Imitation crab sticks	$6$ –12 months at $-18^{\circ}$ C	36	Not available
Surimi	3–5 months at $-18$ °C	36	Not available
Cod	10–11 months at $-20^{\circ}\text{C}$	22, 34, 22	Development of off-flavor, cardboardy,
			Development of
			tastelessness and
			loss of tenderness

the aggregation of collagen observed in hake and trout (*Salmo irideus* Gibb) could be due to the interaction of the molecule with formaldehyde, a product of trimethylamine breakdown (27, 28).

A review on the mechanism of lipid oxidation in seafood was written by Khayat and Schwall (29). They suggested that the lipid oxidation in seafood can be catalyzed by metal ions, and that the oxidized lipids bind with protein to form lipid-protein complexes that are responsible for the changes in quality such as toughening and development of unacceptable flavor and odor. Lipid oxidation is also proven to be temperature dependent. Lipid composition in whole and minced rayfish (*Raja clavata*) muscle was reported to have more significant changes at  $-18^{\circ}$ C as compared to those at  $-40^{\circ}$ C (30). The terms rancid odor and flavor are often used to describe off-odors or flavor notes in seafood products. Rancidity is defined as the objectionable flavor as a result of the accumulation of decomposition products of the lipid oxidation process (31). This rancidity development can also be due to enzymatic lipid peroxidation (32, 33). The enzymic activity in catfish (Ictalurus puntatus) was reported to be temperature dependent and is inhibited at frozen temperatures below  $-10^{\circ}$ C (32). In a study conducted on frozen storage of cod (Gadus morhua), it was concluded that the cold store flavor developed in the fish was due to the lipolysis in the phospholipid fraction to produce hept-cis-4-enal (34). Although generic mechanisms for off-flavor development in frozen fish have been proposed, the process is complex, and therefore individual species have to be looked into (35).

The actual cause of product rejection is variable and complex in seafoods and their products. In our laboratory, it has been observed that the lipid content is not the sole factor that determines the shelf life of fish such as tilapia sp., pangasius, and carp. Besides textural changes such as toughening, sometimes described as the development of fibrous texture, which is commonly associated with frozen fish, characteristic odor and flavor

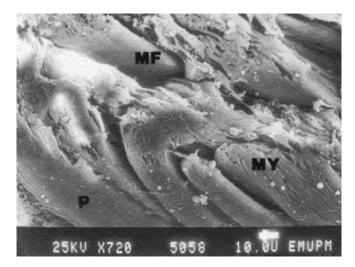
changes have also been noted as causes of rejection. For example, bighead carp frozen for 3 to 4 months developed a strong bitter taste, which has not been noted in species such as tilapia. No reported study on these phenomenal changes has been found in the literature.

In Fig. 4, the microstructure of the white muscle of the bighead carp frozen for 4 months at  $-20^{\circ}$ C is shown. Results of textural evaluation of the cooked muscle showed only an insignificant decrease in the score, although the flavor scope indicated the rejection of the fish.

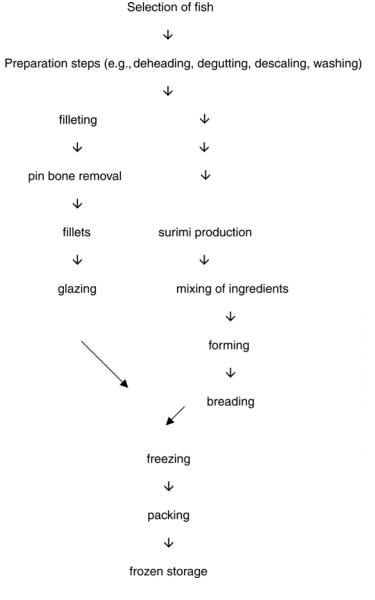
### IV. FREEZING OF FINFISH

Temperate fish such as cod, haddock and salmon are among the well-known commercially frozen species. However, with the depletion of these natural resources, the expansion of aquaculture, and the increase in the acceptability of freshwater fish, frozen freshwater fish such as tilapia, eel, and catfish are entering the cold chain.

Frozen fish are sold as whole, gutted, and filleted and can be frozen in the blast freezer, spiral freezer, tunnel freezer, and plate freezer. Mechanical or cryogenic means can be utilized for the freezing of fish products, but depending on the capacity of the individual manufacturer. Blast freezing on stacked trays may take about 45 min to  $1\frac{1}{2}$ h depending on the size or shape of the product, the targeted temperature, loading, and the capacity of the freezer, among other factors. Blast freezing of precooked fish products such as baked fillets is frequently practiced in the mass catering industry where the incoming product temperature is about slightly above  $60^{\circ}$ C and reaching a final temperature of  $-13^{\circ}$ C. In this case, the product is not followed by a frozen storage. Bulk packing of fillets or gutted fish can also be frozen in plate freezers. Figure 5 shows a simplified flow chart of the processing of frozen finfish.



**Figure 4** The SEM of bighead carp muscle after 4 months of frozen storage at  $-20^{\circ}$ C (P = perimysium; MF = muscle fibres; MY = myofibrils).



**Figure 5** Typical flowchart for the processing of frozen finfish and their secondary products.

#### V. FREEZING OF SECONDARY FINFISH PRODUCTS

Surimi is commonly used as the main raw material for the processing of secondary products, although there are processors who still use minced fish meat alone or in combination with surimi as the raw material. Alaska pollock (*Theragra chalcogramma*) is traditionally the ideal material for the production of surimi; however, with its depleting population, other fish species have been used. Commercially, in the ASEAN countries, threadfin bream (*Nemipterus* sp.), bigeye snapper (*Priacanthus* sp.), jewfish (*Sciaena* sp.), and lizardfish (*Synodus* sp.) have been used to produce surimi of varying grades (36). Elsewhere, attempts to produce surimi from other fish species such as Pacific whiting (*Merluccius productus*), menhaden (*Brevoortia tyrannus*), sardines (*Sardinops* sp.), capelin (*Mallotus villosus*), and herring (*Clupea harengus*), to name a few, have been reported. Surimi blocks of 20 kg wrapped in plastic liners are normally frozen using plate freezers.

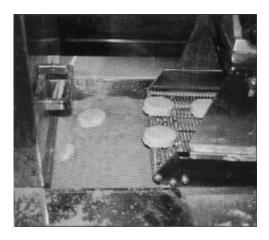
Minced fish-based and surimi based products are popularly distributed frozen (-10 to  $-20^{\circ}$ C) for longer shelf life. Some of the secondary products that are marketed are fish balls, fish cakes, squid balls, shrimp balls, burgers, and sausages. In Fig. 5, the flowchart of the processing of frozen breaded secondary products is shown. Breaded products such as fish nuggets, burgers, and patties are also available. Although better freezing techniques can be used to freeze these products, owing to the economics of production, presently they are blast frozen. Seafood analogs, such as imitation crabmeat, are individually quick frozen (IQF), since they fetch high market prices. The precautions needed for achieving good frozen storage for these products are similar to those already discussed under the subsection of prefreezing and shelf life. A low-temperature environment during processing is also highly recommended to reduce lipid oxidation, since the fish lipid is highly exposed to oxidation during the mixing process (Figs. 6 and 7).

### VI. SAFETY OF FROZEN SEAFOOD AND HACCP

The safety of consumed foods is of paramount importance for the world population today. This is because processed and prepared foods are moved from one part of a continent to another and from one part of the world to another owing to globalization. They are frequently consumed sometime after the actual processing and preparation and kept "fresh" by the application of principles in food science and technology. The concern on the safety of the food is again of utmost importance when the food falls in the high-risk category. In a recent publication, seafood including ready-to-eat fish or fishery products, scombroid toxin-forming species, stuffed seafood products, vacuum or modified atmosphere package fish, and raw fresh or frozen shellfish are listed in the "substantial risk potential" category (40). However, freezing the seafood make it less vulnerable as a source of food pathogens if strict quality control is adhered to and the cold chain has not been broken. As already illustrated in Fig. 1, the cold chain only ends at the point of consumption by the consumers. The risk associated with the consumption of raw frozen seafood could be due to the initial count and type of microbial contamination or the cross-



**Figure 6** Fresh fishball. (Courtesy of SEAFDEC, 1996.)



**Figure 7** Breading of fishburger before freezing. (Courtesy of SEAFDEC, 1996.)

contamination that may occur during postfreezing handling, and it is more serious if the product does not go through terminal heating prior to consumption. During thawing, the product temperature experiences a gradual drop, hence it passes through a temperature range similar to those temperatures of chilled storage, that can harbor a host of pathogenic bacteria. Negligible microbial damage to the frozen food during frozen storage can happen, since at these temperatures growth rates are very slow and generation times may be more than 100 h (41).

Freezing as a preservation technique is intended for an extended storage of the frozen product and hence extreme temperatures below the freezing point of the product are applied. It is noted that microbial growth does not occur at temperatures below  $-6^{\circ}$ C to  $-10^{\circ}$ C. However, microorganisms are different in their sensitivity to these subzero temperatures; some may experience no injury, others only a sublethal injury, and death may occur in yet another (41). Freezing rate, actual storage temperature, and temperature fluctuations to the point of inducing partial melting of ice crystals may determine the extent of damage incurred by the cell membrane of the microorganisms. Improper thawing conditions may provide an opportunity for the revival of surviving microbes. For example,

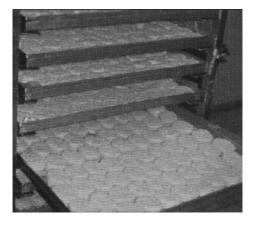


Figure 8 Trays of breaded fish burger ready for freezing. (Courtesy of SEAFDEC, 1996.)

Listeria monocytogenes showed ability to initiate growth in the thawing condition even though the cells were injured during frozen storage (42).

Hazard analysis critical control point (HACCP) is a quality assurance (QA) system to ensure the safety and wholesomeness of the food. Recently, it has been suggested that a QA system should be an integrated system with components of the ISO 9000 and HACCP systems and a comparison of the two quality systems was also made (43). The HACCP system, which is generally agreed upon on a broad base by all countries, consists of the seven basic principles: identification of hazards, determination of critical control points, specification of limits, setting of a monitoring system, corrective actions, verification, and documentation. No specific details are given, although general guidelines are provided, since each HACCP plan is product oriented and again may differ from one processor to another, differences that could be due to a different setup. Currently, HACCP is mandatory for seafood processing plants in the United States (44). Other countries like New Zealand, Australia, and the European Union are also looking into the issue owing to the economics of international trade.

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# 18

# **Freezing Shellfish**

# **Athapol Noomhorm**

Asian Institute of Technology, Pathumthani, Thailand

# Punchira Vongsawasdi

King Mongkut's University of Technology Thonburi, Bangkok, Thailand

#### I. INTRODUCTION

Freezing has become a widely used method for the preservation of shellfish. The need for freezing arose when chilling was not sufficient in keeping shellfish quality for longer storage periods. Good freezing and cold storage enable shellfish to be kept for months or even up to a year or more. Principally, shellfish spoils because of self-digestion and as a result of the action of bacteria. Both self-digestion and the action of bacteria are encouraged by enzymes, which remain active in the shellfish after it dies. Enzyme activity can be reduced by lowering the temperature.

The freezing process alone is not sufficient for preservation. It is merely a method for preparing shellfish for storage at a suitably low temperature. The methods or steps for preparation are solely dependent on the type of shellfish and its anatomy. For example, butchered crabs are cooked before freezing. Oysters are frozen with as well as without shell, different treatments to which are given before freezing. Shrimps are generally blanched in salt solution prior to freezing, and lobsters need to be cooked in brine solution.

The freezing method is the most important factor in the quality of products. The method used should accomplish the freezing process quickly. Freezer design largely depends on the type of product to be frozen, freezing time, and the amount of raw material. A number of freezers or freezing methods are in use: sharp freezing, plate freezing, blast freezing, cryogenic freezing, liquid nitrogen freezing, carbon dioxide freezing, spray or immersion freezing, and pressure shift freezing. Careful selection is needed to keep the product. Each method has advantages and drawbacks, to which a brief discussion is given in this paper.

During freezing and cold storage life, products undergo a complex series of chemical, physical, bacteriological, and histological changes. These changes generally become more apparent after thawing a product. Color, flavor, and texture of the products are the most vulnerable characteristics that are affected by freezing. In shrimp, crab, and lobster, formation of black or blue pigments has been observed. Protein denaturation causes loss of juiciness and results in a poor texture. Similarly, the pleasant taste of shellfish is lost

during storage due to lipid oxidation. These problems can be overcome by providing suitable freezing and subsequent thawing conditions.

# II. SHELLFISH VARIETIES, HARVESTING METHODS, AND PREPARATIONS

### A. Crabs

Crabs are found in the Atlantic and the Pacific. There are two categories: swimming crabs and walking crabs. The dominant varieties in the market are blue crab (*Callinectes sapidus*), stone crab (*Menippe mercenaria*), dungeness crab (*Cancer magister*), Alaska king crab (*Paralithodes camschatica*) and tanner crab (Chionectes spp.). Most of the crab species are captured by single pots. This harvesting method is highly selective, and the products are landed live for maximum quality. The pots are baited with fresh fish and dropped to the bottom of the ocean with a heavy line and marker buoy. The legal size male crabs are daily collected by hauling the pot up from the water. Other gear for harvesting blue crabs are trotline and dredges. The trotline, on which is bait or lures, is dragged behind a vessel as it moves through the water, and used to catch the crabs during the time when the animals are actively feeding. The crabs in a dipnet are collected while the bait is raised to the water surface. The dredge, which has teeth along the bottom bar of a metal frame to dislodge the animals from the bottom, is principally applied during winter. Two dredges are dragged from a boat and hauled alternatively. The fresh animals are well kept before processing (http://www.seafoodhandbook.com/harvest.html).

After unloading, the crabs may be butchered by a stationary iron blade and the carapaces removed. The animals are split in half and cleaned. Both whole crabs and butchered crabs have to be cooked prior to freezing. The crabs are cooked in boiling water or 3% brine at a temperature of 100°C for 20–25 minutes depending on the species and the form of preparation (whole or butchered) (1). In case of blue crabs, cooking is done at a temperature of 121°C for 3–20 minutes. Meat removal of king crabs is done by shaking or blowing with water under pressure and sometimes with rubber rollers, while blue crab meat and dungeness crab meat are usually picked manually.

Crabmeat is delicately sweet, and firm but flaky. It is categorized as white body meat and claw meat. The white meat includes lump, backfin, and flake or regular. Lumpmeat is the finest and most expensive. It consists of large, choice chunks of body meat. Backfin is smaller pieces of the body meat, and flake is white meat from any part of the body in flakes and shreds (http://www.seafoodhandbook.com/harvest/crabmeatforms.html). Cut meat pieces are packed in cartons or trays for freezing. The meat in blocks of cartons is given an ice glaze before packing and shipped to cold storage  $(-18^{\circ}\text{C to } -23^{\circ}\text{C})$ .

### B. Oysters

Oysters differ from the crustacean shellfish in that they contain a significant content of carbohydrate and a lower total quantity of nitrogen in their flesh (2, 3) (Table 1). Three species of oysters are of economic importance, i.e., the eastern oyster (*Crassostrea virginica*), the Pacific oyster (*Crassostrea gigas*), and the Olympia oyster (*Ostrea lurida*). Oysters can be harvested by picking, tonging, and dredging. Picking is generally confined to the area exposed at low tide. Tonging and dredging can capture oysters in larger numbers. Tongs, which consist of two poles crossed like scissors and have toothed iron baskets at the ends of the poles, are lowered to the seafloor and scoop up the animals, and

**Table 1** Nutritional Value of Shellfish (per 100 Grams of Edible Portion)

Item	Dungness crab	Shrimp	Clam	Lobster	Oyster
Calories	94 <sup>a</sup>	84 <sup>a</sup>	64 <sup>a</sup>	80°	90°
Total fat (g)	1.1 <sup>a</sup>	$0.9^{a}$	$0.8^{a}$	1 <sup>c</sup>	$3.2^{\rm c}$
Saturated fat (g)	$0.1^{a}$	$0.3^{a}$	$0.1^{a}$	$0.6-1.9^{d}$	
Monounsaturated fat (g)	$0.2^{a}$	$0.2^{a}$	$0.1^{a}$		
Polyunsaturated fat (g)	$0.4^{a}$	$0.4^{a}$	$0.3^{a}$		
Dietary fiber (g)	$0^{a}$	$0^{a}$	$0^{a}$		
Protein (g)	19 <sup>a</sup>	18 <sup>a</sup>	11 <sup>a</sup>	16.2–21.6 <sup>d</sup>	13.1°
Carbohydrate (g)	$1^a$	$0^{\mathrm{a}}$	$2^{a}$	$0.8^{d}$	
Cholesterol (mg)	65 <sup>a</sup>	166 <sup>a</sup>	$30^{a}$	$60^{\rm c}$	$30^{\rm c}$
Sodium (mg)	322 <sup>a</sup>	191 <sup>a</sup>	49 <sup>a</sup>		68.9–143.4 <sup>b</sup>
Vitamin A (μg)	_	$18-22^{b}$			75 <sup>b</sup>
Niacin (mg)	3.1 <sup>a</sup>	1.58 <sup>b</sup>	_		2.01 <sup>b</sup>
Thiamin (mg)	$0.47^{\rm b}$	$0.034^{\rm b}$			$0.067^{\rm b}$
Riboflavin (mg)	0.167 <sup>b</sup>	$0.034^{\rm b}$			$0.233^{b}$
Vitamin B12 (mg)	$8.9^{a}$	1.3 <sup>a</sup>	43 <sup>a</sup>		
Ascorbic acid (µg)		275–324 <sup>b</sup>		17 <sup>c</sup>	565 <sup>b</sup>
Vitamin E (mg)				60°	1.05 <sup>b</sup>
Copper (mg)	$0.6^{a}$	193.2 <sup>b</sup>	$0.3^{a}$		869.4–1220.2 <sup>b</sup>
Phosphorus (mg)	149 <sup>a</sup>	_	147 <sup>a</sup>		
Selenium (mg)	41 <sup>a</sup>	34 <sup>a</sup>	21 <sup>a</sup>		
Zinc (mg)	4.7 <sup>a</sup>	$0.93^{\rm b}$	_		11.89–14.97 <sup>b</sup>
Iron (mg)	$0.35^{b}$	2.6 <sup>a</sup>	12 <sup>a</sup>		$5.45 - 8.20^{\mathrm{b}}$
Manganese (mg)	16.9–20.9 <sup>b</sup>	33.6 <sup>b</sup>	0.4 <sup>a</sup>		20.8–23.8 <sup>b</sup>

<sup>&</sup>lt;sup>a</sup> From http://www.wholehealthmd.com/refshelf/foods view/0,1523,167,00.html

then are closed before being raised from the bottom (4). Similar to the former gear, a dredge is a metal rake that is dragged across the bottom of the ocean, scraping up oysters in its path. The shellfish are gathered and held in a chain-mesh bag. The oysters are carried up from the bottom either by a conveyor belt or by flowing water through a large diameter hose (5).

The oysters are marketed either in the shell (unshucked) or in the shucked form. If they are marketed in the shell, only washing, packing, and chilling are required. Most oysters, however, are sold as shucked meats, which are prepared by hand labor. After shucking, oysters are rinsed as the pieces of shell and torn or discolored oysters are culled out. Then they are aerated in blowing tanks to remove sand, silt, and shell fragments. The washed meats are size graded and packed into suitable packaging (6). The type of package greatly depends on the freezing method. For example, compression plate freezing is suitable for meats packed in waxed cartons and over-wrapped, while blast tunnels are suitable for meats packed in cans. Freezing rates should be fast, and freezing temperatures should be as low as possible. The recommended temperature is  $-18^{\circ}$ C, although oyster meat may remain in good condition for more than 9 months when stored at  $-29^{\circ}$ C (7). However, storing oyster meat for more than 6 months may degrade raw material quality if the consistency in storage condition is not easily attainable.

<sup>&</sup>lt;sup>b</sup> From Ref. 13.

<sup>&</sup>lt;sup>c</sup> From http://www.charlestonseafood.com/seafoodnutrition.htm

<sup>&</sup>lt;sup>d</sup> From Ref. 3.

### C. Clams

Clams may be hard-shelled or soft-shelled. The edible portion consists of the muscles, the siphon, and the foot. Clams are generally sweet and a bit chewy. Their flavor and tenderness depend on the size and species. Several varieties are available in the markets. Some of them are hard clams or quahog (Venus mercenaria), sea clams or skimmer clams or surf clams, (Spisulla solidissima) and soft clams (Mya arenaria). These bivalves are scooped from the sand at low tide and from beds in deeper water on the Atlantic and Pacific coasts by dredging. Live clams should be tightly closed with a fresh smell. The neck of the soft-shelled clam should retract when touched (http://www.seafoodhandbook.com/ safety/quality.html). After harvesting, the clams are first washed free of sand and silt, and the shell is removed. In some processes, squeezing the meats eviscerates the clams, which removes the stomach and other soft tissue. The shucked animal should not dry out, shrivel, or discolor. Excessive or cloudy liquid and shell particles should not be found. Clams may be chopped, sliced into strips or left whole before packing and freezing. Like other shellfish, storage and freezing types are also dependent upon the type of container used. Smaller packages (2 kg) are best frozen by the compression plate method, and larger cans should be frozen by either blast or shelf coil methods.

# D. Shrimp

More than 300 species of saltwater and aquacultured shrimp are marketed worldwide. Saltwater shrimp are categorized as warm and cold water species. Warm water shrimp, classified by shell color (white, pink, and brown), are harvested in the south Atlantic and the Gulf of Mexico. These are *Penaeus setiferus*, *P. aztecus*, and *P. brasileinsis*. (http://www.ncfisheries.net/kids/3shrimp.htm). Coldwater shrimps, which possess firmer meat and sweeter flavor, are caught in the North Atlantic and the northern Pacific. These are *Pandalus borealis*, *P. dispar*, *P. goniurus*, *P. platyceros*, and *Cragon franciscorum augustimana*. The sea shrimp are captured by a trawl net from the stern of the trawler. The trawl is a large funnel-shaped bag held open by otter boards for entrapping the shrimps. However, this gear is quite low in specificity to the animal. Therefore considerable time is needed to separate the shrimp from other sea fishes (http://www.seafoodhandbook.com/harvest.html).

Aquacultured shrimps, especially black tiger shrimps (Penaeous monodon), are commonly raised in Asia. In 1996, the world shrimp production was around 3 million tons. According to the FAO, Thailand is the top farmed shrimp producer, while China and India maintained production levels of around 80,000 and 70,000 tons, respectively (http://www.fao.org/waicent/ois/press\_ne/presseng/1997/pren9735.html). Another species of shrimp is the blue-legged shrimp or giant long-shrimp (Macrobrachium rosenbergii). This species is indigenous to the Indo-Pacific region and can be sorted into eight categories according to market desire and physical stages, i.e., (1) large, (2) medium, (3) small shortclaw males, (4) long-claw males, (5) females without and (6) with eggs, (7) soft shell or newly molted, and (8) terminal growth males. The common gear used for harvesting these aquacultured shrimp are the cast net, the haul net, the large dip net (9), and sometimes electrical gear (10). Two harvesting techniques are applied, i.e., cull (continuous) harvesting and drain (batch) harvesting. In cull harvesting, the shrimp are captured by single-seine operation. Five to six workers at different locations in the pond beat the water surface and walk in the same direction as the seine is being pulled. The advantage of this practice is that the small-sized shrimps are returned to the pond. In batch technique, the water is drained through a water gate behind which a bag net is fixed. Shrimps swept along the stream of water are collected in this net. Drain operation is usually applied when the shrimp attain market size or when the cessation of farming activities is forced by a lack of water or a fall in the temperature (11). Those live shrimp are immobilized or chill-killed by being dipped in ice water or ice brine (3% salt) slurry and then packed single-layered on ice. This method can prevent the shrimp from damaging one another and retard tissue degradation (12).

Shrimp contain low fat and few calories, but they are higher in cholesterol than most seafood (13) (Table 1). Fresh shrimp have a clean smell, with no trace of ammonia or indole. The meat should be translucent and dense (http://www.seafoodhandbook.com/safety/quality.html). If caught many miles from shore, the shrimp are usually beheaded before the vessel reaches dockside. This saves considerable space in the bins, because only the tails of the shrimp are the edible portion (6).

After unloading from the vessel, the packing ice is removed and the shrimp are conveyed to a rotary drum to remove surplus water and debris. They are weighed and graded according to size. For peeled and deveined products, the shrimp with shells on are peeled and deveined by hand or by a mechanized process. After peeling, the tail meats are washed and inspected. They may be blanched in salt solution for about 10 minutes and dried to remove excess water prior to freezing. Four common forms of shrimp are prepared for the frozen markets. They are frozen headless, frozen peeled and deveined, uncooked frozen and breaded, and headed and unshelled (http://www.seafoodhandbook.com/harvest/shrimpforms.html).

A temperature of -32 to  $-40^{\circ}$ C is recommended for freezing shrimp (1). Such low temperatures are achievable by using blast or multiple freezers. At lower storage temperatures, the development of a rancid flavor is minimized.

### E. Lobsters

The lobster, the king of crustaceans, provides sweet, firm, and succulent meat. The live lobster has a hard and intact shell (http://www.seafoodhandbook.com/safety/quality. html). When lobster is lifted, its tail curls under. Two species of lobster are common in the market, i.e., American lobster and rock lobster. The American lobster or true lobster or Northern lobster (*Homerus americanus*) is caught from Maine, while the rock lobster or spiny lobster (*Jasus lalandii*) is found off Florida's west coast, Southern California, and the Pacific, or from Australia or New Zealand. The difference between those species is that the rock lobster lacks large claws but has long spiny antennae or feelers. Three rock lobster species are marketed worldwide, i.e., *Panulirus argus*, *P. interruptus*, and *P. cyanus*. The lobsters are caught by either baited trap (pot) or trawler. The trap, which consists of an oblong box made of laths or wood slats spaced to allow the undersized animals to escape, are used to gather the inshore lobster. The latter gear is suitable for deep-sea lobsters inhabiting water of up to 200 fathoms deep.

Various practical methods are used for the preparation of lobster prior to freezing. Likely it needs to be cooked in 3% brine for 10 to 20 minutes. However, the heat requirement of deep-sea lobster is only to cook the meat next to the shell but not the meat below the surface to avoid sticking of the meat to the shell; otherwise, electric shock will be applied. For the spiny lobster, the preparation steps include breaking the tail from the body for the removal of the intestine.

Lobster meat is packed in cans for freezing. The normal package size in practice is 14 to 16 oz. Best results are reported when lobster meat is frozen stored at  $-23^{\circ}$ C or lower, with an intended shelf life of 3 months.

### III. FREEZING METHODS

Freezing and subsequent cold storage is considered an excellent process for preserving the qualities of shellfish up to 18 months or more. During this period, a complex series of chemical, physical, bacteriological, and histological changes are retarded. There are various freezing methods employed based on raw material and available resources. Following are the factors that should be considered while selecting the suitable method (5):

- 1. Type of product to be frozen
- 2. Allowable freezing time for products of average weight
- 3. Handling requirements
- 4. Source of available power
- 5. Space requirement
- 6. Amount of raw material
- 7. Cost of equipment and operation

# A. Sharp Freezing

Sharp freezing is generally considered as a slow freezing method because it takes 3-72 hours, depending on the amount of product to be frozen. The procedure consists of placing the products in a very cold room where temperatures are maintained in the range of -20 to  $-40^{\circ}$ C. The disadvantages of this method are its low freezing rate and its high labor cost. In addition, the cooling coils may frost during the loading and unloading of the products. Therefore defrosting is required at least once every six months. A sharp freezer consists of an insulated room with multishelf racks for holding the product. The shelves are stacked one above another. Each shelf is constructed of pipe formed into a horizontal flat coil. Refrigerant, especially ammonia, is expanded through the coils lowering their temperature to the desired level.

### B. Plate Freezing

Plate freezing is accomplished by direct contact between a cold plate and the products. Pressure applied to the plates on each side of the products improves contact and increases the heat transfer coefficient between plate and products. The pressures applied are between 1 and 10 bar using hydraulic pressure. Since the pressure exerted is constant, some expansion takes place as the product freezes. However, owing to the pressure, expansion takes place inside the package until all voids are filled. Approximately 7% expansion occurring in the products during freezing is sufficient to fill voids and compress the products into single block during freezing. There are two types of plate freezer, the horizontal plate freezer and the vertical plate freezer. Both types will efficiently freeze only regular-shaped packages or blocks. The plates are made from extruded aluminum, which, in cross section, shows channels through which the liquid refrigerant is passed. Horizontal plate freezers are often used to freeze prepacked retail flat cartons of shrimp (both shell on and shell off). The products are usually wrapped in plastic film and then packed in cartons

or directly onto aluminum freezing trays, which are, in turn, placed on the freezer shelves (14). Plate freezing is also applied to spiny lobsters and clams.

## C. Blast Freezing

The blast freezer utilizes very cold air (0 to  $-40^{\circ}$ F) for removing heat from the products and transporting it to the refrigeration coils. There are several types of blast freezers. Although the general operating principles are the same, airflow, loading method, and capacities vary widely. Most blast freezers use average air velocities of 2.5 to 7.5 m/s while 2.5 to 5.0 m/s are reported as the most economical velocities. Two important modes for blast freezing are tunnel freezing and fluidized bed freezing.

In the former method, the products are placed on trays, either loose or packaged. The trays are placed on a moving mesh belt passing through a tunnel or enclosure where the cold air is blown from opposite end. In some designs, cold air is circulated on both top and bottom ends of the entire length of the freezing belt so that a better distribution of the cold air is obtained.

The main advantage of tunnel freezers is their versatility. They are suitable for irregular-shaped, different-sized, and nondeformable foods such as crustaceans, fish fillets, and added-value products. However, tunnel freezers have a slightly slower freezing rate than immersion freezers, and dehydration may happen to the products during the operation, which results in the constant need of defrosting the equipment. To reduce moisture loss from the products, two or more stages of freezing are introduced. When large volumes of air of high relative humidity are applied in the first stage, the products are frozen with a minimum water loss. In the later stage, the temperature differences and the vapor pressure differences are not as great. Therefore the cold air has a substantially less desiccating effect.

In some plants, where high capacity or extended freezing times are required, the conveyor length become excessive. This problem has been overcome by the use of multiple-pass systems where the products are transferred inside the freezer from one belt to another, and travel backwards and forwards along the length of the freezer (14). A more usual method of overcoming the same problem is to use a spiral freezer. The single continuous belt can be operated on a single- or a twin-drum application in ascending or descending combinations. The whole system is enclosed in an insulated chamber. Blast freezing is suitable for many aquatic food products, for example, king crab in the shell packed into trays or cartons, whole dungeness crab in cans, and peeled and deveined shrimp on thin aluminum sheets.

Owing to the high demand for individual quick frozen (IQF) products, fluidizing belt freezers are extensively used. The procedure requires a sufficiently powerful stream of cold air to keep the products in suspension. The great advantage of fluidized bed freezing is short freezing times, since each piece of food is kept loose and free flowing by the air pressure, resulting in a higher yield. The retention time for the freezing operation depends upon the size of the products, for example, small shrimps require 6–8 min while the large sizes require 12–15 min. Examples of fluidized bed freezers are the Freez-Pak fluidized belt freezer and the Lewis fluidized bed freezer (5).

## D. Cryogenic Freezing

Cryogenic freezing is the ultrafast freezing process that results in excellent product quality. In this method, the products either unpacked or thinly packed are exposed to an extremely

cold refrigerant. The heat removal is accomplished during a change of state by the refrigerant. The advantages of cryogenic freezing are rapid rate of freezing, simple, flexibility, and inexpensive equipment design. Refrigerants commonly used in plants are liquid nitrogen or carbon dioxide (14).

## 1. Liquid Nitrogen Freezing

Liquid nitrogen is a by-product obtained during the production of oxygen from air. It is nontoxic and relatively cheap. The critical point for nitrogen occurs at  $-147^{\circ}$ C and 3.39 MPa. The triple point occurs at  $-210^{\circ}$ C and 12.6 kPa. Liquid nitrogen freezing systems are divided into three types: the immersion type, the spraying of liquid nitrogen type, and the circulation of very cold nitrogen vapor type. However, a spray of liquid nitrogen is commonly applied in food industries. In this system, the products are placed on a conveyor belt in a single layer. The conveyor carries the products through the freezer in the opposite direction to the flow of nitrogen. Warm products entering the freezer are first subjected to a blast of cold nitrogen gas (typically at about  $-50^{\circ}$ C). This precooling stage can prevent stress cracking in the products as a result of too rapid a cooling. Later, the products are headed to the direct application of liquid nitrogen, which has a boiling temperature of  $-196^{\circ}$ C at atmospheric pressure. The final freezer section allows the temperature gradient from the outside to the center of the products to reach equilibrium (15).

Shrimp and oysters are successfully frozen by this procedure. It is found that the frozen product obtaining from liquid nitrogen freezing provides lower indole and trimethylamine content than those obtaining from conventional methods (5). In addition, smaller ice crystals are formed, and less protein changes. These result in less drip loss during thawing.

### 2. Carbon Dioxide Freezing

Liquid  $CO_2$  can exist only at the critical temperature ( $-31^{\circ}C$ , 7.35 MPa absolute pressure) and the triple point ( $-56^{\circ}C$ , 7.35 MPa absolute pressure). Freezing with carbon dioxide is done by passing the products under specially designed nozzles. Liquid  $CO_2$  supplied to the nozzles at about 300 psi is sprayed toward the products as they move under the nozzles on a conveyor belt. The  $CO_2$  changes state as it leaves nozzles and absorbs large quantities of heat from the products.

At atmospheric pressure and room temperature, solid  $CO_2$  (dry ice) converts directly from the solid to gaseous state (sublimation) leaving no liquid residue. As sublimation of dry ice occurs at  $-78.5^{\circ}C$ , it is possible to freeze to at least  $-75^{\circ}C$  (15). Freezing under these circumstances is very rapid, and drip losses are reduced to less than 1% (14). Using dry ice in cryogenic freezing is a thermic process. The operation involves mixing of the comminuted dry ice with the products in the interior of a slowly rotating and insulated drum. The rotation of the cylinder not only turns the products over and over in the dry ice but helps to break up the gas film on the products as well. It is noted that all of the dry ice must be separated from the frozen products before the products are packed, otherwise the packages may explode owing to the pressure of the gaseous  $CO_2$  in the headspace (5).

### E. Immersion Freezing

Direct immersion means immersing the products into a low temperature liquid such as sodium chloride brine, sugar solution, or glycerol. Sodium chloride, which has a eutectic point of  $-212^{\circ}$ C, is normally applied in the freezing process at the temperature of about

 $-15^{\circ}$ C. Further reduction in temperature must be achieved by transferring the products to cold storage. The limitation of immersion freezing is the suitability of the refrigerating medium (14). The products should be edible and capable of remaining unfrozen at  $-17.8^{\circ}$ C and slightly below. The refrigerating temperature also needs to be carefully controlled. If the temperature is too high, the medium will enter the products by osmosis; if too low, the medium may freeze the products. Moreover, it is not easy to maintain the medium at a definite constant concentration (5). Both crab and shrimp can be frozen by brine immersion. Cooked whole and eviscerated crabs in the shell are dipped into a circulating brine of 88° salometer at -18 to  $-15^{\circ}$ C for 45 min and then brought into fresh cold water to remove excess brine and provide an ice glaze (14).

# F. Pressure Shift Freezing

This technology permits achievement of a uniform supercooling in the whole volume of the products. Thus a significant improvement of quality in terms of ice crystals can be obtained. Pressure shift freezing is carried out in a high-pressure vessel whose temperature is controlled at subzero temperature. The products are firstly refrigerated under pressure, and no ice crystals are formed in this step. The pressure is then released to atmospheric pressure. This phenomenon causes three significant phases. The first phase corresponds to cooling down the products without phase change. In the second phase, the temperature suddenly rises up to the phase change temperature at the current pressure. Finally, partial freezing is initiated owing to the high supercooling of the product (16).

### IV. GLAZING

Glazing is the process of coating a frozen product with a layer of ice to retard moisture loss and oxidation rate. Glazing is usually accomplished by dipping the frozen products into a tank of chilled water or by spraying a light coating of chilled water onto the frozen products. The low product temperature causes a coating of ice to form on the product exterior. The amount of ice per product can be obtained by controlling the temperature of the frozen products and that of the glazing water as well as the residence time in the glazing tank. Generally, a dipping tank provides the possibility of building up bacteria, so the spray system provides some advantages over the former system. However, many different additives can be applied in the chilled water e.g. the following (17):

- 1. Organic salt solution of disodium acid phosphate, sodium carbonate, and calcium lactate
- 2. Alginate solution or Protan glaze
- 3. Antioxidants such as ascorbic and citric acids, glutamic acid, and monosodium glutamate
- 4. Other edible coatings such as corn syrup solids

The advantages of the additives are preventing oxidation, improving the appearance of the products, and strengthening the ice layer so that it will not be so brittle as to fracture when the products are bumped or dropped (18).

During storage, evaporation occurs from the layer of glaze only. Glazed products have a shelf life of at least 6 months, while products without a protective covering last only 3–4 months. Another preferable technique is to rely on a moisture-vapor-proof overwrap

on the product packages. This ensures preventing water loss during prolonged frozen storage as well.

### V. THAWING

Proper thawing is as important as the selection of a suitable freezing method because it can affect the net weight of the products. Improper thawing under forced conditions of warm air or water may cause the products to release natural juice, thus drying out the products and inviting bacterial growth. Therefore frozen products should be thawed slowly at temperature just above freezing as in cold water (<a href="http://www.seafoodhandbook.com/harvest/frozen.html">http://www.seafoodhandbook.com/harvest/frozen.html</a>.). Special equipment has been devised for the purpose. The frozen blocks are thawed by dielectric heating, being conveyed on a rubber belt through a series of dielectric units. To get an even heat flow across the frozen pieces, blocks are first immersed in plastic trays of water to fill up the voids in the blocks. It takes one hour to thaw a 4 inch thick block (1). Alternatively, cross-flow air blast devices are also used to thaw frozen blocks.

# VI. EFFECTS OF FREEZING, FROZEN STORAGE, AND THAWING ON COLOR, FLAVOR, AND TEXTURE

Changes in color, flavor, and texture occur immediately after harvest. If not properly handled during freezing, frozen storage, and thawing, the shellfish may undergo quality changes, making the products unacceptable to markets and consumers. Following are the main types of quality changes.

# A. Color Changes

In shrimp, the rapid formation of black pigments, widely known as melanosis, occurs within a few hours after death and is enhanced by exposing the shrimp to air. This reaction is the result of phenol oxidation in the internal shell surface and can occur within 2–12 hours of exposure even at  $0^{\circ}$ C (5). However, at  $-18^{\circ}$ C, no visible spots were detected during 3 months of storage (14). The discoloration starts at both ends of overlapping shell segments and then it develops into black bands or a zebralike appearance. In an advanced stage, the oxidation reaction of tyrosinase on tyrosine results in melanin pigment on the underlying shrimp meat. Copper and other metallic irons can accelerate the reaction. Thawing also influences the appearance of the frozen animal. When shrimp are thawed at a temperature higher than  $0^{\circ}$ C, melanosis may occur owing to the unnecessary exposure of the shrimp to air, thus leading to oxidation.

In crab and lobster, the development of blue or black discoloration or blueing is one of the most troublesome problems. Blueing may occur after freezing or during frozen storage, or it may appear after thawing and subsequent air exposure or even shortly after cooking. This bluish-black curdlike discoloration appears to be related to biuret-type reactions between the copper pigments in the circulating fluid and the heat-denatured protein. As cited by Babbitt (19), blueing relates to the change of phenolic compounds in crab as well. Tyrosinase and phenol oxidase in live crabs initiate an oxidation reaction, and then the nonenzymatic oxidation and polymerization occur afterwards, particularly under alkaline conditions and in the presence of metals such as copper and iron. The

molting stage can aggravate the incidence, since the phenolic compounds are involved in the formation of the new shell. Banks et al. (5) noted that the blueing discoloration in king crab meat could be reversed by using a reducing agent such as sodium sulfite solution. Babbitt (19) suggested that the oxidases in crab are inhibited by antioxidants like ascorbic acid and metal chelating substances like phenylthiourea. Heating the crabs at 100°C for 20 minutes completely inactivates all enzyme activities. However, the best way to reduce the blueing discoloration is processing only live crabs, which experience proper harvesting and handling.

Other discoloring problems found in crab and lobster meats are yellowing and fading of the red or orange—red carotenoid. Both indicate a degree of oxidation during processing or long cold storage, which depends upon the retention time of exposure to air and the temperature, freezing condition, and storage condition. Microorganisms also cause discoloration in some shellfish; for example, Asporogenous yeast can grow and produce pink pigment when contaminated in oyster (2).

# B. Free Liquor or Drip

Free liquor or drip usually occurs when the frozen products are thawed. Cloudy liquid is originally attributed to the rupturing of cell walls caused by ice crystal formation during freezing. It has been postulated that drip or exudate formation is directly related to the capacity of the animal protein to hold moisture (14). During cooking, there will be an increase in the release of watery cook liquor resulting in the loss of water-soluble proteins, particularly sarcoplasmic protein, vitamins, and minerals. This exudate indicates inappropriate handling, prolonged ice storage prior to freezing, inappropriate cold storage temperatures, or improper thawing. For example, frozen oyster shows a drip loss of over 20% depending on the conditions of blowing (5).

### C. Texture Changes

Frozen shellfish gradually loses its juiciness and succulence after freezing and subsequent frozen storage. Such textural changes are caused by protein denaturation. Frozen shellfish muscle loses all its moisture easily during the first bite and therefore subsequent chewing results in a very dry and slightly tough texture; This is also true for crab, shrimp, and lobster when stored for prolonged periods. Species and storage temperature are the main factors affecting the change in texture, for example, dungeness crab meat is less juicy than king crab meat when kept at  $-18^{\circ}$ C. Lower storage temperatures can also improve the keeping quality of aquatic animals.

Some species of shellfish experience another type of serious textural change. The mushiness or softening of the flesh is found in several species of aquatic animals. These include sand crab (20), rock lobster (21), and blue legged shrimp (22). This texture deterioration is due to the proteolysis of digestive enzymes from the hepatopancreas (20, 23). The microstructure changes of the animal are related to the development of mushiness through the use of the scanning electron microscope (SEM) and the transmission electron microscope (TEM). The disintegration starts from the perimysium, the endomysium, the z line, and the H zone, together with the degradation of connective fibers and sacroplasm (24). Mushy shellfish is externally indistinguishable from the fresh animals; the condition becomes evident only after cooking. Poor handling can diminish shellfish qualities by accelerating the rate of degradation. Blanching can lessen the problem, because enzymes are inactivated at temperatures higher than 70°C. Crab should

be cooked for 8 to 10 minutes (20), while the blanching condition of shrimp is 65°C for 15 to 20 seconds (12). However, the animals may lose their juiciness owing to water loss during the operation. Beheading is the other way to diminish the enzyme problem, especially in shrimp, since the hepatopancreas, the major source of digestive enzymes, is removed. The disadvantage of beheading is that the product possesses less flavor because of the removal of the hepatopancreas, which is the main source of flavor as well (25). The molting stage is also a crucial factor affecting the degree of mushiness. Pre- and postmolt shrimp are mushier owing to a larger proportion of short fiber than at the intermolt stage (20, 26). Moreover, the animal begins to absorb water upon entering the premolt stage. Such water may soften the tissue by disintegrating the interfiber connection within the muscle. After molting, the amount of water in the tissues gradually decreases, but it is enough to cause significant myofibrillar disruption and consequently mushiness (26).

# D. Changes in Odor and Flavor

Shellfish has a mild and sweet taste, with a pleasant aftertaste. However, this specific characteristic is lost quickly when stored under unsuitable conditions. Generally odor changes occur in three phases, i.e., gradual loss of flavor due to loss or decrease in concentration of some flavor compounds; the detection of neutral, bland, or flat flavor; and the development of off-flavor owing to the presence of acids and carbonyl compounds from lipid oxidation and the degradation of trimethylamine oxide (17).

Flavor and odor components found in shellfish are mostly classified in the nitrogenous compound group. These compounds comprise free amino acids, lowmolecular-weight peptides, nucleotides, and organic compounds. Shrimp and crab possess high levels of taurine, proline, glycine, alanine, and arginine, but only traces of peptides are detected. Nucleotides serving as important taste producing factors are found in shellfish as adenosine monophosphate (AMP). Small amounts of adenosine diphosphate (ADP), inosine monophosphate (IMP), guanosine monophosphate (GMP), and uracil monophosphate (UMP) are detected in the leg meat extracts of boiled crab as well (27). Trimethylamine oxide (TMAO) is a common base usually found in the muscle of fish and shellfish. In the postmortem stage, this compound is reduced from trimethylamine, which provides fish odor by bacterial strains of Enterobacteriaceae including Escherichia coli, Achromobacter, Micrococcus, Flavobacterium, nonfluorescent Pseudomonas, Clostridium, Alcaligenes, and Bacillus spp. (28). As cited by Konosu and Yamaguchi (27), TMAO is detected in crab and shrimp in the range of 65–140 and 172–213 mg/100 g, respectively. The other important quaternary ammonium base is glycine betaine found in crab and shrimp in amounts of 357–711 mg/100 g and 251–961 mg/100 g, respectively. The variations depend upon species, growth, freshness, parts and tissues, season, and environmental conditions. Other decomposition odor in shrimp is indole. This strong odor is a result of highly proteolytic indole positive bacteria such as Aeromonas and Proteus spp. These microorganisms attack muscle protein and convert tryptophan to indole. The reaction is aggravated when the shellfish is stored under high temperatures; therefore a high level of indole can be used as indicative of temperature abuse (29).

# VII. MICROBIOLOGICAL QUALITIES

Freshly caught shellfish are highly perishable owing to bacterial activities, so the animals are preferably either frozen or boiled as soon as possible after capture. The incidence and

number of microorganisms greatly depend on the quality of water from which these animals are harvested. The initial flora found in freshly caught oysters are Alcaligenes, Flavobacterium, Moraxella, and Acinetobacter spp., while shrimp, crab, and lobster have a bacteria-laden slime on their body surfaces including Bacillus, Micrococcus, Pseudomonas, Acinetobacter, Moraxella, Flavobacterium, Alcaligenes, and Proteus spp. (30). However, when the shellfish are frozen, these microorganisms are generally inactivated. Thus, during frozen storage, microbiological changes in shellfish tissue are usually minimal. The microorganisms that undergo very low temperatures can be characterized as uninjured, injured, or killed (31). Although some microorganisms survive, their activities are suppressed, and bacterial numbers may be considerably reduced if the recommended temperature is maintained. The temperature below which microbial growth is considered minimal ranges from -10 to  $-12^{\circ}$ C (32). However, the surviving microorganisms, usually psychrotrophic bacteria, e.g., Pseudomonas, Acinetobacter, Moraxella, Alcaligenes, and Flavobacterium spp., may grow after thawing if time permits and and thus can lead to microbial spoilage of the thawed products (30, 31) (Table 2). The microbial activities depend on the degree of freshness of the raw material, the natural microflora in the shellfish tissues, and the thawing technique used.

**Table 2** Microbial Spoilage of Some Shellfish

Shellfish	Microorganism	Reference
Shrimp	Acinetobacter	30
1	Moraxella (at 5–11°C)	30, 31
	Vibrio	30
	Pseudmonas (at 0°C)	30, 31
	Proteus (at 16–22°C)	31
Crab meat	Pseudomonas	30
	Acinetobacter	30
	Moraxella	30
	Proteus	30
Raw lobster	Pseudomonas	30
	Alcaligenes	30
	Flavobacterium	30
	Bacillus	30
	Vibrio (including Vibrio parahaemolyticus)	30
Oyster	Pseudomonas	2, 30
	Acinetobacter	2, 30
	Moraxella	2, 30
	Serratia	2
	Proteus	2
	Clostridium	2
	Bacillus	2
	Escherichia	2
	Enterobacter	2
	Flavobacterium	2
	Lactobacilli	2, 30
	yeasts	2, 30

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# 19

## **Freezing Secondary Seafood Products**

#### **Bonnie Sun Pan**

National Taiwan Ocean University, Keelung, Taiwan

#### Chau Jen Chow

National Kaohsiung Institute of Marine Technology, Kaohsiung, Taiwan

#### I. INTRODUCTION

Fishing including postharvest handling and processing is a primary industry worldwide. The fish processing industry consists of micro, small, and medium-sized businesses whether in remote fishing ports or in metropolitan areas. Their primary objective is to preserve the freshness and safety of seafoods, since these are quickly perishable after catch. The industry used to serve as an intraregional supplier of fresh produce with extended shelf life and later expanded into global sourcing and marketing for a wide spectrum of seafood products. The nature of the commodities evolved from fresh produce of a low profit margin to value-added secondary products using innovative technology. The key technologies required for the transformations are the means to increase yield, energy conservation, labor efficiency, mechanization or automation, product stability, and versatility in forms and functions (1).

#### II. FROZEN SASHIMI FISH

Sashimi fish have the highest value among different categories of seafoods. In Japan, a variety of saltwater fish is used for sashimi and sushi including tuna (maguro), marlin or sailfish (kaziki), mackerel (saba), flounder (hirame), squid (ika), prawn (ebi) and exotic species like abalone (awabi), octopus (tako), and sea urchin roe (uni).

#### A. Tuna Sashimi

Sashimi fishes have the highest value among different categories of seafood, while tuna sashimi has the highest economic value of all fishes. Tuna is priced at auction depending on the fishing gear of the catch, the on-board freezing temperature, and the fat content of the fish. Sashimi-grade tuna is either iced or frozen on-board at -50 to -60°C. Those frozen stored at -20°C are for canned tuna and not for sashimi use.

#### 1. Market Demand

Bigeye, yellowfin, bluefin, and southern bluefin tuna harvested by ship and frozen stored aboard at  $-50 \sim -60^{\circ}$ C provide tuna of sashimi grade. The only global market for sashimi tuna is Japan. The demand is about 450 thousand tons a year. The major suppliers besides Japan are Taiwan and Korea, having market share of 26% and 13%, respectively in 1999. Southern bluefin is the most valuable tuna, followed by bluefin. The import prices are more than double those of the bigeye, and four to five times those of yellowfin (2).

In recent years, around 40,000 tons of tuna are air-freighted fresh to Japan from a wide range of countries to supply the sashimi market demand (3).

#### 2. Histamine Defect

Biogenic amine content and especially histamine content in tuna need monitoring. Histamine is the main cause of scombroid poisoning, which is associated with the migratory fishes from the families of *Scomberosocidae* and *Scombridae* that have high levels of free histidine in their muscle tissues (4, 5). Histamine and other biogenic amines are found at very low levels in fresh fish and are later developed by the contaminated bacterial flora having positive decarboxylase activities (6–12). Temperature effects on histamine decarboxylase and histamine formation exhibit parabolic responses (13). The use of nomographs based on time-temperature history (10) and Arrhenius plots can estimate histamine formation and shelf life (14). Data on the production of histamine by bacteria, and on their measurement, and predictions of the growth have been published (15).

The Food and Drug Administration has established a defect action level of 50 ppm in tuna and other fish species (16).

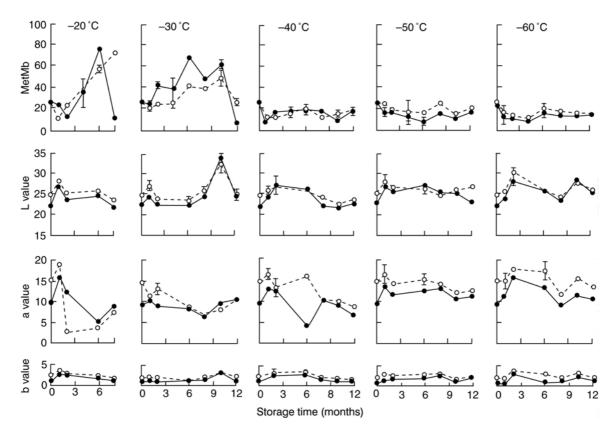
#### 3. Postharvest Handling

Tuna harvested by pole and line are for sashimi, while those from purse seiners are for canning because the former have higher operational cost and provide better quality catch. Tuna catches are bled and graded on board into three categories: live, prerigor, and full rigor. Devices are used to reduce tuna struggling in water and on board in order to prevent internal bleeding, which appears as dark red stains in muscle and causes the sashimi tuna to depreciate in value. The dressed tuna are weighed, quick frozen, glazed, and stored at  $-50^{\circ}$ C or below on board (17).

## 4. Discoloration

a. Oxidative Discoloration. The color of the muscle affects the value and the way of using the tuna. The fresh muscle of albacore appears pinkish in color and turns to white after cooking. It has been used for canned products. Tuna that has red muscles and a relatively high fat content are used for sashimi, e.g., bluefin tuna and southern bluefin tuna.

Tuna regardless of their species are susceptible to discoloration (Fig. 1) during storage at  $-20^{\circ}$ C for a period of  $2 \sim 6$  months (18–20). Lowering the storage temperature to a range between  $-35^{\circ}$ C and  $-78^{\circ}$ C effectively retards the oxidation of myoglobin (Mb) and hemoglobin (Hb), thus preventing discoloration (18). The method of thawing affects the color appearance of tuna. Thawing in running water results in less oxidation of Mb than air thawing and microwave thawing (21). The frozen-thawed meat undergoes discoloration more quickly than unfrozen tuna during the subsequent iced storage



**Figure 1** Changes in MetMb% and Hunter's L, a, b values of bluefin tuna cubes during frozen storage for 12 months. The fish cubes were excised from fresh fish followed by freezing at  $-60^{\circ}$ C for 36 h, and then stored at -20 to  $-60^{\circ}$ C, respectively. MetMb% were measured immediately before thawing, while Hunter's L, a, b values were measured after thawing at  $4^{\circ}$ C. The surface and inner portions of the fish cubes were examined. Symbols:  $\circ$ , surface portion;  $\bullet$ , inner portion. The bars represent the standard deviations (20). (From Ref. 20, with permission.)

(19, 21, 22). Rate of tuna discoloration measured as MetMb/total Mb is dependent on the temperature of frozen storage (20, 23–25).

- b. Silver Sheen. A silver sheen covers a localized surface of the red muscle of tuna. It is caused by the accumulation of excessive lactic acid produced from anaerobic glycolysis during struggling. The extent of sheen may spread as time proceeds after catch, and it reduces tuna's suitability for sashimi (25).
- c. Pale-Soft-Exudative Meat. This type of discoloration is generally found in tuna caught from waters of low latitude and relatively high water temperature. The pale discoloration occurs under the skin in a localized area that may expand into deeper sections of tuna.

The texture of this kind of pale tuna meat is soft and exudative or watery. It is not suitable for sashimi or sushi.

The cause of this phenomenon is the struggling of tuna while hooked to the longline. The ATP hydrolyzes to supply energy for muscle contraction and produces inorganic phosphate in addition to the lactic acid formed from anaerobic glycolysis. These acids accumulate in muscle and result in the dropping of pH to  $5.8 \sim 5.9$ . At this pH range, Mb is vulnerable to oxidation and sensitive to temperature fluctuation even around  $0^{\circ}$ C (25). The pale-soft exudative (PSE) tuna is indeed similar to the PSE of pork, and is

undesirable. The precautions in postharvest handling are also similar to those of PSE pork.

- d. Blood Stain. The struggling of tuna on deck causes internal bleeding especially in tail muscle owing to its exhausting motions. Such blood stains in meat reduce the value of sashimi (25).
- e. Burn. When live tuna is put directly into cold water for storage on board, the temperature change results in burn in the muscle. It appears as if the tuna had been treated with high temperature or cooked. The fresher the fish, the more severe burns appear in muscle (25).

#### 5. Preventing Discoloration

a. Proper Handling. Preventing the fish from struggling when hooked or on deck reduces or eliminates the discoloration. Keeping the live fish from seeing light lessens the excitement of fish.

Spiking the spinal cord of tuna destroys the brain response center of heat generation that keeps the body temperature at 27°C against the low temperature of the fish hold.

Bleeding quickly and gutting on board before storage slows down the rate of oxidation and deterioration. Immersion of tuna in precooling tank helps the body temperature to drop down to 0 to  $-0.3^{\circ}$ C prior to frozen storage. Proper handling methods to preserve freshness in tunas have been compiled by the Japan Fish Conserving Development Association (25).

b. Color Fixation with CO. Carbon monoxide (CO) readily binds myoglobin covalently and forms a stable cherry-red pigment, carboxymyoglobin (MbCO), proven to extend shelf life of prepackaged fresh beef (26). CO has much stronger binding affinity toward Mb than O<sub>2</sub> does. The partition constant is 250 times different (27). Treatment of the fresh cut tuna with CO prior to vacuum packaging helps to retain the bright red color by forming MbCO.

In commercial practice, sashimi tuna steaks and tilapia fillets undergo color fixation by flushing 100% CO into plastic bags inside of which the cut fish are stacked in meshed trays. After the bag is filled with CO, it is tied and stowed at 4°C for a period of time. It takes 24 h for tuna steak 2 cm thick, and  $30 \sim 40 \, \text{min}$  for tilapia fillet, to have the surface color fixed (28). The residual CO in tuna reaches  $0.9 \sim 1.0 \, \text{mg/kg}$  (29) and loses 50% during iced storaged for 1 day. It further decreases to  $0.06 \, \text{mg/kg}$  after 7 days. Frozenstored tuna steaks maintain 50% of the residual CO at the surface after 2 months and changes very little inside the tuna steak even after 6 months (30).

The presence of CO reduces the oxidation of Mb and keeps the MetMb below 10% in tuna during frozen storage (28). However, Japan has issued regulations (31) for inspection of the CO residue in fish using the gas chromatographic method and forbids the CO treatment of fish, while the Norwegian authority permits the use of up to 0.5% CO in the modified atmosphere for meat treatment (32).

c. Frozen Storage. During frozen storage at  $-20^{\circ}$ C, CO-treated bigeye tuna steak has a much slower rate of Mb oxidation. MetMb% remains unchanged below 10% for 6 days, while the untreated tuna steak oxidizes rapidly forming  $40 \sim 60\%$  MetMb in 6 days (28, 30).

The temperature of frozen storage results in differences in discoloration in tuna at storage temperatures between -40 and  $-60^{\circ}$ C. In this temperature range, formation of MetMb and discoloration are negligible. Storage at -20 to  $-30^{\circ}$ C results in noticeable

increases in MetMb% and discoloration indicated by Hunter color values of L, a, b (Fig 1) (20).

Proper packaging materials must be used to protect frozen seafoods from dehydration and oxidation during freezer storage. Vacuum packaging in moisture-proof plastic film works well for white fish or shrimp but is not suitable for sashimi tuna or tilapia or milkfish fillet that consist of red muscle fibers. The low oxygen tension in the vacuum package turns the bright-red MbO<sub>2</sub> into the deoxy form. The meat appears magenta in color. At low oxygen tension, Mb is more readily oxidized to form MetMb than at ambient environment. The MetMb is brownish in color, which does not appeal to consumers.

## B. Tilapia Sashimi

Tilapia (*Oreochromis spp.*) originated from Africa has become an important cultured hybrid food fish. Major suppliers of the cultured tilapia are Taiwan, China, Thailand, and Indonesia. In addition, the Philippines is promoting tilapia cultivation using idled shrimp ponds (33). The production of this cultured fish in Asia has grown rapidly. For example, in 1953, Taiwan produced about 6,000 tons of tilapia at an average price of about USD 0.11/Kg (NTD 4.61/Kg), in 1999 about 57,000 tons was produced and averaged USD 1.0/Kg (NTD 30.91/Kg) (34). The ninefold increase in average price was caused by the development of frozen sashimi tilapia.

Tilapia each 1 pound or larger are cultured in salt water prior to harvest. The fish are chilled in ice water, filleted, skinned, deboned, and then washed and soaked in salt water (1–2%) for 3–5 min followed by ozone treatment, color fixation with CO, vacuum packaging, liquid nitrogen freezing, and frozen storage. All frozen tilapia processing plants have to comply with good manufacturing practices (GMP) and hazard analysis and critical control points (HACCP) (28).

The freezing rate of tilapia chunk correlates (r=0.99) with freezing temperature ranges -7 to  $-128^{\circ}$ C regardless of freezing method (35). However, the muscle structure is maintained better with liquid nitrogen freezing at -87 and  $-128^{\circ}$ C than with airblast freezing at -20 to  $-35^{\circ}$ C. The differences in ultrastructure of tilapia frozen at various temperatures disappear during prolonged storage at  $-20^{\circ}$ C. The shelf life for high structural quality of tilapia is predicted to be 2.7 months of storage at  $-20^{\circ}$ C for tilapia frozen with liquid N2 (36). However, the discoloration of frozen tilapia fillets appear along the lateral line much sooner than the dorsal or ventral part.

The original tilapia introduced to Taiwan was *O. mossambica* and later *O. zilli* and *O. nilotica*. They were consumed as fresh fish or frozen round fish, marketed among low-value fishes. The developments of monosex culture of all male hybrid fish, the color fixation of the fillet with CO, and the superchilling of the fillet as frozen sashimi turn this fish into an elite secondary seafood. It is now named Taiwan Tilapia, and it has another name in Chinese meaning "tidal porgy" or "tidal sea bream" to distinguish it from the original tilapia. This marketing strategy of building a new image attached to a transformed commodity using innovative technology has been legendary.

#### C. Unconventional Fish Sashimi

Wild and cultured salmon have been gaining popularity among Asian sashimi consumers. The high fat content, the texture, and the color cause salmon sashimi to soar in Asian markets, mainly in Japan followed by Taiwan. The major suppliers are Norway, Iceland,

Canada, and the U.S.A. More recently, Chile and New Zealand have started to export salmon for sashimi use.

Cobia (*Rachycentron canadom*) cultured in the marine cage net has become a new species for sashimi use. The one-year-old fish weighs 6–8 kg, and the two-year-old fish reaches 15 kg. The fat content in dorsal meat is 16–26%, and in ventral meat 20–28%. The high fat content meets the requirement of sashimi fish. The shelf life of cobia sashimi is 6 days at 4°C and 12 h at 25°C (37).

#### III. FROZEN SHRIMP AND PRAWNS

Shrimp and prawns are regarded as among the most valuable seafoods in international trade. The leading suppliers include China, Thailand, and the U.S.A., while the major consumers are in Japan, the European Union, and the U.S.A. The demand for individually quick frozen (IQF) products is higher than that for block frozen products.

## A. Glazing

During frozen storage, the quality of shrimp and prawns is degraded by oxidation, denaturation, dehydration, and recrystallization. Ice-glazing is applied to protect the frozen shrimp and prawns from these undesirable quality changes.

The glaze content normally ranges 8–12%, but ice coating as thick as 25–45% can be found commercially (38). Introducing standardized glazing procedures and complying to a regulated glazing content are important to the producers and consumers. The glaze content is commonly determined by CODEX procedures using a gravimetric method. An enthalpy method has been developed applicable to an automatic control system for glazing of prawns. It is a noncontact measurement of glazing percentage using infrared thermometry (38–40). In addition, federal inspection and grading standards on minimal meat content must be compiled with.

## B. Biochemical Indices for Choosing Freezing Methods

During cold storage of shrimp or prawns, the amount of volatile basic nitrogen (VBN) increases with storage time in a pattern similar to the microbial growth curve consisting of a lag phase and a log phase. The breaking point happens to be at 25 mg per 100 g, which is coincidentally the quality standard for beheaded–peeled shrimp. The content of trimethyl amine oxide (TMAO), and the ratio of TMAO to TMA, decreases linearly with storage time (41). Thus TMAO, the ratio of TMAO to TMA, and VBN and indole can be used to characterize quality and to predict shelf life for chilled or frozen shrimp (42).

Air blast freezers including IQF or contact freezers are generally used in the frozen seafood industry. In recent decades, liquid nitrogen freezers have been applied for the processing of high-value cultured aquatic products. In order to evaluate the effects of freezing method and frozen storage on prerigor grass prawns, the spacing between muscle fiber bundles and the catheptic activity of the intact lysosomes recovered from the frozen shrimp are suggested as better indices than salt soluble protein extractability. Liquid nitrogen freezing maintains better muscular and cellular integrity than air-blast freezing indicated by the ultrastructure of muscle and catheptic activity of the ruptured lysosomes (43).

#### C. Black Discoloration

Marine crustaceans are vulnerable to black spot development during chilled and frozen storage. Early studies show that black discoloration appears in the homogenate of blood and liver of lobster while the tyrosinase activity is higher in blood than in other organs (44, 45).

Polyphenol oxidases have been identified and purified from shrimp (46–49), crab (50), krill (51), and lobster (52–55) catalyzing the melanosis. These polyphenol oxidases are converted to active form from the latent form, prophenol oxidase, by trypsin (53–56), which is abundant in the hepatopancreas of shrimp originally for digestion (57).

However, polyphenol oxidase from hemocytes is unstable and is inactivated in a few days at temperatures below 4°C, while black spots develop rapidly in frozen storage of thawed prawns (58). Hemocyanin, an oxygen carrier/storage protein, being at high concentration in plasma, is converted to phenoloxidaselike enzymes with similar biochemical properties of the propolyphenolase. The phenoloxidaselike enzymes are stable for more than a month during frozen storage and are likely potent inducers of black spot development in prawns (59).

In an attempt to keep the natural color of shrimp, industries have used inexpensive additives, e.g., sodium bisulfite or metabisulfite. Shrimp are dipped in a 1.25% solution for 1 min. Labeling is required for this chemical treatment due to the health hazard to consumers with asthma. The control level for residual additive is 100 ppm. Safer alternatives have been tested. For example, kojic acid and 4-hexylresorcinol, commercially known as Clean Cruster and Everfresh<sup>TM</sup>, and sodium pyrosulfite are effective inhibitors of black discoloration. Vitamin E and catechinic acid are not effective in preventing the discoloration in shrimp (60).

#### IV. FROZEN PREPARED SEAFOODS

## A. Roasted Eel

The major market for frozen roasted eel (*Aguilla japonicus*) is Japan. It has been a custom for generations that Japanese eat roasted eel on July 3, which is called *Wusinohi*, Ox Day. The Japanese believe eel is very nutritious. Consuming eel helps one to recuperate from summer exhaustion. In light of this tradition, export of frozen roasted eel from Taiwan peaks in June and reduces afterwards in a yearly cycle.

Live cultured eel are processed into frozen roasted eel. At the processing plant, eel are graded by sizes of 3, 4, 5 or 6 eel to a kg and transferred to holding tank unfed for 1 to 2 days to reduce the possible muddy odor. It is caused by geosamin accumulation in muscle during culturing in ponds overpopulated with phytoplanktons or algae. The live eel are again transferred into iced water 5–8°C or below to put them to dormancy for 30 min or even 2 to 3 h, followed by bleeding, gutting, washing, and filleting. Fillets may be cut into three pieces. Pairs of the same cut are stretched with 3 bamboo sticks, then roasted as a popular form of consumer product. Whole fillet can be roasted as another form of product.

Based on type of seasoning, there are mainly two kinds of products: *sirayaki* is the kind without seasoning, while *kabayaki* is with seasoning. In addition, eel viscera are stretched with bamboo sticks and roasted as *kimoyaki* (61). The eel fillets are roasted on a meshed conveyor through tunnels with a gas flame under the conveyor, for roast eel to reach a center temperature of 60–65°C followed by conveying to a second tunnel for

steamed eel to a center temperature of 80°C. The eel fillets are then soaked in preheated soysauce-based seasoning and kept at 80°C followed by a second seasoning at 40°C for eel center temperature to decrease to 60°C. The seasoned fillets are then conveyed through a second tunnel to be roasted to a center temperature of 82–86°C followed by another seasoning at 70°C and again roasted for a third time in tunnel (62).

The finished roasted eel are precooled to below  $10^{\circ}\text{C}$  and moved into an air-blast freezer with a spiral conveyor for IQF or into a liquid nitrogen freezer for better quality. The center temperature is further reduced to below  $-18^{\circ}\text{C}$ . The products are packaged in polyethylene and put into wax-coated corrugated boxes or styrofoam boxes for frozen storage at temperatures generally below  $-25^{\circ}\text{C}$  by air blast.

By-products of eel processing, such as the bone oil and eel calcium, are manufactured and sold as nutraceuticals or dietary supplements for increased profitability.

Applying the same processing to roasted milkfish belly flap gives a new seafood product for the Taiwan market.

#### B. Breaded Seafood Products

#### 1. Breaded Shrimp

Butterfly and popcorn shrimp are frozen prefried products with added value. They are prepared by thawing of frozen shrimp, soaking in 10% brine with crushed ice and then draining, or by using fresh shrimp. The shrimp are then beheaded and peeled into the butterfly form, battered, predusted and breaded, and prefried at  $190^{\circ}$ C for about 20 seconds. They are then cooled to room temperature, IQFed to a center temperature below  $-18^{\circ}$ C, packaged, and stored at a freezer temperature below  $-25^{\circ}$ C.

#### 2. Breaded Fish Steaks or Fillets

Breaded restructured fish steak is made from fresh or frozen fish. The quality of the fish steak depends greatly on the freshness of the raw material. If frozen fish is used, thawing has to be done in a cold room to reduce thaw drip. The fish is filleted and skinned, deboned, chopped, washed, dewatered, mixed with surimi, seasoned at  $\leq 5^{\circ}$ C, and mixed under vacuum (0.8–0.9 bar) for a total mixing time of 10–15 min followed by chilling to a center temperature of  $-2^{\circ}$ C. The mixed mince is restructured with a forming machine using compressed air, and then it is battered, predusted, breaded, prefried at 180–190°C until the crumb appears golden brown in color; then it is IQFed to a center temperature of  $-18^{\circ}$ C or below and stored at air temperatures of  $\leq -28^{\circ}$ C. Breaded cuttlefish or squid steaks are produced by similar processes.

Erobed product of catfish is processed through filleting, coating with seasoning of an oil-base mixture, and freezing (63).

The coating of this type of product cannot exceed the flesh content. A void between shrimp or fish meat and batter indicates either that the batter formulation or the battering method is not adequate and should be avoided.

## 3. Breaded Surimi Products

Products like analogs of crableg, scallops, shrimp, minced fish burgers, minced fish balls, minced fish roll, minced-fish-coated squid sticks for gumbo, and breaded chopped steaks of fish or squid are popular frozen surimi products.

## C. Ready-to-Serve Gourmet Seafoods

Foods such as shrimp dumplings, salted clams, squid salads, and seasoned kelp salad are manufactured as chilled and ready-to-serve products for use in homes and restaurants.

Retort pouch foods have been marketed as shelf-stable convenience foods and found their major market in Japan, where the annual consumption exceeds 200,000 tons. A manual on retort pouch seafood has been prepared that includes formulation and processing technology for shellfish, fish, and squid, e.g., sweet–sour prawns and prawns in tamarind sauce (64).

#### V. SAFETY AND QUALITY

## A. Temperature Control

Food deterioration is linked to temperature. Chilled foods, especially seafoods, are vulnerable to temperature fluctuation in storage. The temperature is monitored starting from the raw material handling stage, and through processing, packaging, and storage into distribution systems to manage product safety and to preserve restaurant quality. By monitoring the temperature, the shelf life can be predicted, and lot-to-lot consistency can be assured. Thermographic imaging of food within display cabinets offers rapid assessment of the food products rather than measuring the air temperature, which may bear a vast disparity from product temperature. H-E-B Food and Drug Co. (San Antonio, TX) has developed a system to measure, monitor, and control food temperature and has installed it in retail distributors' premises (65).

## B. Disinfection of Foodborne Pathogens

Unsatisfactory hygienic conditions in raw material handling and processing result in food poisoning outbreaks. Since sashimi and sushi undergo no heat treatment throughout the preparation, disinfection procedures are incorporated to assume hygienic quality and food safety. Ozone treatment has been used for this purpose. Immersion of fish fillets into ozonated water for less than a minute greatly reduces the bacterial counts. The extent of reduction is dependent on ozone concentration and treatment time (66, 67). Ozonolysis of bacterial DNA, i.e., that of *E. coli* and in phage M13 outside of bacterial cells, occurs within 30 min of immersion of shrimp meat in 5 ppm ozonated saline (68).

#### C. Quality Assurance

Establishing specific quality indicators for different fish products is very important. The color and the tone of tuna meat are good indicators for determining freshness. The MetMb/total Mb, K-value, pH, TMA-N, TMAO/TMA ratio, and VBN are chemical properties related to freshness or spoilage. K% is the hypoxanthine and inosine versus the total of ATP and all the derivatives. It can be as low as zero for prime quality tuna and 10–20% for sashimi grade; further increases means degradation in sensory value and eating quality (69).

The hygienic quality of seafood products needs special attention. Sashimi and sushi have to comply with the microbiological guidelines for ready-to-eat foods. APC and total *E. coli* are indicator organisms of hygienic quality. Specific pathogens including *Vibrio* parahaemoliticus, *Listeria monocytogenes*, *Staphylococcus aureus*, *Salmonella* species, and

Shigella species have been found in sashimi and sushi and have caused gastrointestinal illness. The VBN of sashimi should be below 15 mg/100 g.

Fillets frozen prerigor appear fresher than those frozen postrigor after the fish fillets are thawed. The difference decreases with prolonged freezer storage (70).

Twice freezing should be avoided for seafood products. The formaldehyde content increases in double frozen pollack (*Pollachius virens*) fillets. Sensory evaluation shows increases in firmness and fishy odor, and decreases in freshness, after twice freezing (71). Color of the refrozen fillet turns to more yellowish and reddish as shown by increases in both +b and +a values.

In coated or breaded shrimp or fish products, the actual percent fish flesh (DPFF) has to be tracked through the processing system and weighed. The AOAC has official methods for measuring both APFF and DPFF (72).

#### VI. CONCLUSION

The market demand for seafood is sensitive to economic conditions and consumers' interests. The affluent society shows more concern on health than the average, implying a growing market sector for healthy and high-quality foods. The demographic shift to a greater ratio of older population to the younger also creates a special market sector. This sector of people traditionally consumes more seafood. The growing interest of these two sectors of consumers in health issues drives the food consumption pattern to demand more seafood of better quality. It is foreseeable that the global market for seafoods will increase.

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# **20**

## **Frozen Seafood Safety and HACCP**

## Hsing-Chen Chen and Philip Cheng-Ming Chang

National Taiwan Ocean University, Keelung, Taiwan

Finfish and shellfish are the major dietary protein sources next to meat and poultry for most of the world. Since their flesh is composed of soft tissue with high moisture and free amino acids as well as water extractable nitrogenous compounds, seafood are highly digestible and nutritious. However, under condition of mishandling, microorganisms can easily proliferate in seafood.

In 1997, the global total landing of seafood was close to  $1.2 \times 10^8$  MT. About 40% of this harvest goes into the international trade, and the total export value has increased from \$33,000 million (U.S.) in 1990 to \$54,500 million in 1996. The share of developing countries in total fish exported expanded during the 1980s and 1990s to reach around 50% in 1997. The EU, Japan, and the United States imported around 75% (in value) of the internationally traded fish (1). People worldwide enjoy fresh, processed, and even raw seafood. However, because of mishandling, outbreaks of seafood-borne illnesses occasionally occur.

In this chapter, microorganisms that are found to exist intrinsically in seafood, and their effects on food safety, are described first. Since to prepare food free from infectious microorganisms is the responsibility of food processors, the Hazard Analysis Critical Control Points (HACCP) for frozen seafood processing are then mentioned.

## I. FROZEN SEAFOOD SAFETY

## A. Microorganisms in Seafood

The predominant microflora found in fresh finfish and shellfish are shown in Table 1. There is some diversity in microorganisms between these two groups of seafood. Distributions of microorganisms in seafood are influenced by many factors. Spoilages of seafood are mainly related to the amounts and varieties of putrefactive strains contaminating the foods. The distribution of microorganisms in the body of a fish is also different.

#### 1. Factors Affecting the Microbial Diversity in Finfish

Variations of microflora in finfish are influenced mainly by their eating habits and living environments. In the same water area, different species of fish may harbor different

**Table 1** The predominant Microorganisms Found in Fresh Finfish and Shellfish

Seafood	Microorganism
Finfish	Acinetobacter, Aeromonas, Alcaligenes, Alteromonas, Bacillus, Corynebacterium, Flavobacterium, Micrococcus, Moraxella, Proteus, Pseudomonas, and Vibrio
Shellfish	Acinetobacter, Aerobactoer, Aeromonas, Alcaligenes, Arthrobacter, Bacillus, Candida, Clostridium, coliforms, Corynebacterium, Flavobacterium, Lactobacillus, Micrococcus, Moraxella, Moraxella-Acinetobacter, Proteus, Pseudomonas, Rhodotorula, Sarcina, Staphylococcus, Torulosis, Trichospora, and Vibio

Source: Modified from Ref. 2.

microflora, because of their ingestion of different food. Since seawater sustains a diversity of microorganisms, in particular *Moraxella*, *Corynebacterium*, *Acinetobacter*, *Vibrio*, *Flavobacterium*, *Pseudomonas*, and *Photobacterium* (2–4), the microbial flora found on seafood are primarily affected by the seawater and geography. Cold marine water fish, for instance, carry mainly psychrophilic gram-negative bacteria such as *Moraxella*, *Acinetobacter*, *Pseudomonas*, *Flavobacterium*, and *Vibrio*, while warm marine water fish harbor numerous gram-positive mesophilic bacteria such as *Corynebacterium*, *Bacillus*, and sometimes enteric bacteria (5, 6).

The bacterial counts of fish also vary with different methods of capture. Trawled fish usually carry bacterial loads 10- to 100-fold higher than those of periods of time along the sea bottom prior to landing (2).

#### 2. Putrefactive Capacity of Microorganisms

Microorganisms capable of producing hydrolytic enzymes (e.g., protease, lipases, and DNAase) degrade seafood more easily. Some species of *Pseudomonas* and *Alteromonas* exhibit strong spoilage activity, while *Moraxella, Acinetobacter*, and *Alcaligenes* are moderately active. *Acinetobacter, Lactobacillus, Flavobacterium, Micrococcus, Bacillus*, and *Staphylococcus* show a low spoilage activity and then only under specific conditions (2). Seafood with different compositions exhibits different tendencies in microbial degradation. Using the growth of *Pseudomonas* as an index in five species of seafood at 35°C, crab is the most susceptible to microbial spoilage, followed by mackerel, cuttlefish, sword shrimp, and pomfret, in descending order (6). In a systematic experimental procedure for fish shelf life modeling to predict the quality of fish in the chill chain (0 to 15°C), pseudomonads are also a good spoilage index (7).

#### 3. Distribution of Microorganisms in Body of Finfish

Microbial levels in different parts of the fish are varied; in general, skin has  $10^2-10^7$  CFU/cm², intestinal fluid has  $10^3-10^8$  CFU/mL, and gill tissue has  $10^3-10^6$  CFU/g (2). Actually, these variations are attributed mainly to water conditions and temperature. Finfish and crustaceans from colder ( $<10-15^{\circ}$ C) waters generally have counts of  $10^2-10^4$  CFU/cm² on skin and gill surface, while animals from warmer waters have  $10^3-10^6$  CFU/cm². Tropical shrimp carry higher numbers of bacteria,  $10^5-10^6$  CFU/g, than cold water species,  $10^2-10^4$  CFU/g. Counts for intestinal contents vary widely from as low as  $10^2$  CFU/g in nonfeeding fish to  $10^8$  CFU/g in actively feeding species. Counts in mollusks also show great variation with water temperature and extent of pollution, from  $<10^3$  CFU/g in cold

unpolluted water to > 10<sup>6</sup> CFU/g in warm waters in which bacterial pollution levels are high (8). Microflora can also vary among different fish species. The predominant bacteria in the intestinal tract of carp include *Aeromonas hydrophila*, *Bacteroides* type A, *Citrobacter freundii*, *Pseudomonas*, and *Micrococcus*, while those in tilapia are mainly composed of *Bacteroides* type A and B, *Plesiomonas shigelloides*, and *A. hydrophila* (9). In addition, the same fish reared in seawater or freshwater can harbor different microorganisms in the intestine. For instance, the dominant genera in the intestinal content of salmon reared in fresh water include *Aeromonas* and Enterobacteriaceae; while *Vibrio* predominates in salmon reared in seawater (10).

#### 4. Microflora in Shellfish

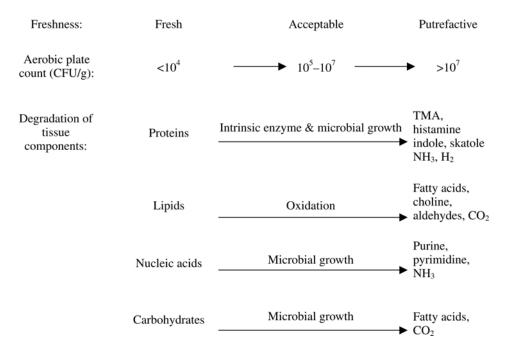
There are two groups of shellfish, crustaceans (crab, shrimp, lobster, crawfish, etc.) and mollusks (bivalves, squids, snails, etc.). The predominant bacterial flora in fresh hardshell shrimp (*Metapenaeopsis barbatus*) harvested from the Taiwan Strait are *Acinetobacter* (33%), coliforms (11%), and *Vibrio* (7%) (11). On the other hand, the natural microbial flora of freshly caught Georgia Coast shrimp are *Acinetobacter*, *Enterobacter*, and *Flavobacterium* (12). Shrimp unloaded from the trawler have an average bacterial count of  $6.0 \times 10^5/g$ , and market shrimp,  $3.2 \times 10^6/g$ . Bacterial counts used for shrimp quality indicator are  $1.3 \times 10^6/g$  (acceptable),  $1.1 \times 10^7/g$  (fair), and  $1.9 \times 10^7/g$  (poor) (5).

In the United States, the hemolymph of about 20% healthy blue crabs from Chincoteague Bay is found sterile according to tests carried out on 290 freshly caught crabs (13). Higher bacterial floras are found in the blue crabs from the Columbia River, USA, in water close to human habitation. The gills of crabs are heavily contaminated by bacteria  $(10^3-10^7/g)$ , when compared to  $1 \times 10^{-4} \times 10^2/g$  in muscle tissue (5).

Being filter feeders, bivalves pass a larger volume of water through gills to obtain oxygen and food. Particulate matter, including microorganisms, are trapped on the gills, transferred to the mouth, and finally digested. Since bivalves sometimes rear and live in estuarine areas where waters are contaminated with sewage, pathogens are occasionally found in them (14). Mussels harvested from approved shellfish water in the Adriatic Sea were examined for the presence of *Vibrio*, *Salmonella*, *Campylobacter*, and verocytotoxin producing *Escherichia coli* (15).

## B. Seafood Safety

Degradation of seafood after harvest begins with enzyme reactions; the intrinsic enzymes in tissue decompose macromolecules such as proteins, glycogen, and nucleic acids into small molecular substances available for microbial growth. The proliferation of microorganisms results in further decomposition and the subsequent production of simple derivatives of tissue substances and of metabolites such as trimethylamine (TMA), fatty acids, aldehydes, ketones, ammonia, and carbon dioxide (Fig. 1). The rate of degradation is temperature dependent. The oxidation of lipids in flesh occurs even at low temperatures when the activity of microorganisms is almost completely inhibited. Some of these products have an off-flavor and/or are toxic. These substances in food, after ingestion, will sometimes cause illness. In addition, the consumption of seafood contaminated with pathogens and/or the toxins produced by them is hazardous.



**Figure 1** Correlation of freshness, microbial spoilage, and chemical degradation of seafood after harvest. (From Ref. 2.)

## 1. Microorganisms Related to Seafood-Borne Diseases

Some pathogens of seafood-borne illnesses, excluding cases of paralytic shellfish poisoning, are illustrated in Table 2.

A. hydrophila occurs in freshwater, sewage, and brackish water and is a common contaminant of fresh foods, including fish and other seafoods (16). It is evident that this species is a human pathogen associated mainly with diarrheal symptoms. This species is predominant among suspected foods, including prefrozen or inadequately cooked seafood and oysters (17). The organism is psychrotrophic with its minimum growth temperature ranging from -0.1 to  $1.2^{\circ}$ C (18). From fish and shellfish, Hänninen et al. (19) identified Aeromonas spp. from 93% of fish samples, 100% from fish eggs, 16% from shrimp, and 100% from freshwater samples. They found that A. hydrophila hybridization group (HG) 3 was predominant in fish, fish eggs, and freshwater samples.

Clostridium botulinum is derived most commonly from sediments and can be assumed to be present on whole fish. Type E and nonproteolytic strains of types B and F can be isolated from the intestine and occasionally from the skin of marine fish. In animals raised by aquaculture, conditions resulting from poor management practices aggravate the occurrence of pathogenic microorganisms, e.g., increased incidence of C. botulinum (8). C. botulinum type E is a psychrotroph, which can produce toxin above 10°C under anaerobic conditions (20). Most pretreated seafood products are distributed and stored frozen and are packaged under modified atmospheres involving CO<sub>2</sub>, N<sub>2</sub>, or vacuum packaging and oxygen-absorbent technology. When the products are subjected to temperature-abusive storage conditions, growth of proteolytic strains of C. botulinum may occur. Non-proteolytic strains of C. botulinum have been found to produce toxins at a temperature as low as 4.4°C (21).

Listeria monocytogenes was considered as a food-borne pathogen after contaminated coleslaw was identified as the vehicle of infection in an outbreak of listerosis in 1981 (22),

**Table 2** Pathogens of Seafood-Borne Illness Excluding Cases of Paralytic Shellfish Poisoning

Pathogen	Major vehicle seafood			
Aeromonas hydrophila	Shellfish (oyster, clam)			
Campylobacter spp.	Processed seafood			
Clostridium botulinum type E	Semipreserved seafood			
Escherichia coli	Processed seafood			
Listeria monocytogenes	Raw fish, shellfish			
Pleisiomonas shigelloides	Cuttlefish, raw oyster, salted mackerel			
Shigella spp.	Raw fish, shellfish			
Salmonella spp.	Processed seafood			
Staphylococcus spp.	Processed seafood			
Vibrio alginolyticus	Raw or undercooked shellfish			
V. cholerae O1	$SAA^\mathrm{a}$			
V. cholerae non O1	SAA			
V. fluvialis	Shellfish (oyster, clam, shrimp, crawfish)			
V. furnissii	SAA			
V. hollisae	SAA			
V. mimicus	SAA			
V. parahaemolyticus	Raw seafood			
V. vulnificus	Raw or undercooked mollusks			
Hepatits A	Shellfish			
Norwalk virus	Shellfish			

<sup>&</sup>lt;sup>a</sup> Same as above.

Source: Modified from Ref. 2.

though it has been recognized as a human pathogen since 1929. It has been isolated from fecal specimens of healthy animals and people, as well as from sewage, silage, fertilizer, vegetable matter, and many foods (23). Important characteristics of this bacterium are capable of growth at 1–45°C, pH 4.3–9.5, water activity of 0.90 or higher, and in salt concentrations higher than 10%. Although several types of food products have been involved in cases of listerosis, the involvement of fish and fishery products is still uncommon. Seafood-related listeroses are those of lightly preserved products such as smoked fish products, marinated products, or raw shellfish (24, 25).

The incidence of salmonellosis has been increasing over the past 50 years (26), but few *Salmonella* outbreaks associated with fish or shellfish are documented in the literature. An outbreak of *Salmonella* in the United Kingdom associated with a fish-and-chip shop was linked to a food handler who was a pathogen carrier (27). Two successive outbreaks of *Salmonella* were caused by consuming improperly prepared chilled, boiled salmon, which affected 87 people (28). The British Surveillance Group within the Public Health Laboratory System reported the incidence of *Salmonella* in 22 of 566 raw shellfish examined (29). A survey of 331 food samples including 55 seafood products in the Malaysian marketplace reported a 25% (4 of 16 samples) incidence of *Salmonella* in raw prawns (30). The field laboratory of the U.S. Food and Drug Administration collected and tested 11,312 imported and 768 domestic seafood samples from 1990 to 1998 for the presence of *Salmonella*. It was reported that the overall incidence of *Salmonella* was 7.2% for imported and 1.3% for domestic seafood, and nearly 10% of imported and 2.8% of domestic raw seafood products were positive for *Salmonella* (31).

Mesophilic Vibrio spp. have been isolated from both pelagic and bottom-dwelling fish. Among the potentially pathogenic Vibrio occurring naturally in finfish and shellfish, V. parahaemolyticus is most widespread. The mesophilic Vibrio spp. are most commonly found in inshore waters with reduced salinity. For example, V. vulnificus is commonly found in estuarine fish, particularly in bottom feeders, but is less common in offshore fish (32). Vibrio is the genus most often implicated in diseases of bacterial origin resulting from eating contaminated shellfish (33). V. parahaemolyticus has often been held responsible for food poisoning (34). V. cholerae non O1 has been found to be involved in choleralike infections of the intestinal tract and other systems (35). The other halophilic Vibrio spp. such as V. mimicus, V. fluvialis, V. hollisae, V. damsela, and V. alginolyticus are often involved in both gastrointestinal illness and septicemia (36, 37). The presence of Vibrio spp. in seafood products is common. Baffone et al. (38) examined fish caught from the Adriatic Sea and shellfish harvested from coastal water of the area of the Adriatic Sea. They found those seafood products were contaminated with halophilic vibrios belonging to the species V. alginolyticus (81.48%), V. parahaemolyticus (14.8%) and V. cholerae non O1 (3.7%). Thus in Western countries seafood-related illness caused by pathogenic Vibrio spp. is commonly associated with crustacean or molluscan shellfish, while finfish are a common vehicle for outbreaks in Japan and other Asian countries.

There is no apparent correlation between *Vibrio* levels and those of fecal indicator organisms, *Escherichia coli*, enterococci, fecal coliforms, or total coliforms (39). *E. coli* O157:H7 is a member of the enterohemorrhagic group of pathogenic *E. coli*. The organism has contaminated a wide variety of foods including meat, milk, fruit juice, and vegetables (40). Although it is rarely isolated from seafood, with its tolerance to acidic and dry environments (41) and its resistance to freeze—thaw operation (42), *E. coli* O157:H7 exhibits a potential hazard to acid-treated seafood and frozen seafood under mishandled conditions. *Campylobacter jejuni* causes bacterial enteritis in humans. Food vehicles often identified in outbreaks are poultry and raw milk. Campylobacteriosis from seafood is also related to poor food handling during processing.

Viruses are not associated with food spoilage, since they are obligate intracellular parasites. They may survive well in food following contamination. Seafood may be contaminated with indigenous marine viruses, which are the most abundant life form in the sea (ca.  $1 \times 10^{10}$  particles/liter) (43). However, only contaminant human viruses have ever been associated with illness in seafood consumers. Viral problems are therefore limited to the role of food in recycling human viruses back to humans (44). In this case, viruses can contaminate seafood via contamination at source, via sewage pollution of the marine environment, and via inadequate hygienic practices of operatives or systems. A number of human viruses transmitted by the fecal—oral route are associated with disease in shellfish consumers. Such viruses include caliciviruses, astroviruses, rotaviruses, adenoviruses, enteroviruses, and hepatitis A virus. Recent taxonomic proposals classify the human enteric caliciviruses into two genera, Norwalklike viruses and Sapporolike viruses (45). The largest reported outbreak of shellfish-borne hepatitis A involved almost 300,000 cases in Shanghai, China in 1988 and was attributed to clams (46).

Parasites are most often animal host–specific and can include humans in their life cycles. Parasitic infections are commonly associated with undercooking products or cross-contamination of ready-to-eat food. Fishborne parasites in products that are intended to be eaten raw, marinated, or partially cooked can be killed by effective freezing techniques. Parasites can occur extensively in finfish and crustaceans, but few of the many helminthic

parasites are capable of infecting humans. They rarely cause illness except where fish are eaten raw or after mild processing (47). Freezing followed by frozen storage will normally destroy almost all fish parasites dangerous to humans. This procedure is recommended for raw products to be eaten as sashimi (raw fish slices). Freezing does not affect marine toxin accumulated in the living animal nor bacterial toxins produced during improper storage before freezing.

## 2. Food Safety of Raw Seafood

In certain areas of the world, some fresh seafood is eaten raw, such as sashimi and oysters. Since seafood, especially shellfish, may contain a variety of pathogens including both indigenous and extraneous species, the ingestion of raw seafood imposes a threat to consumer health. Some indigenous pathogens found include *Vibrio*, *Clostridium botulinum* type E, *Aeromonas*, and poisonous phytoplankton, while extraneous pathogens include *Salmonella*, *Shigella*, *Listeria monocytogenes*, *Campylobacter jejuni*, *Staphylococcus aureus*, *E. coli*, *Bacillus cereus*, Hepatitis A, and Norwalk virus. Food-borne illness resulting from eating raw seafood is sometimes caused by *V. parahaemolyticus* and *V. vulnificus*.

In Taiwan, tuna, oil fish, swordfish, cuttle fish, and purplish amberjack (*Sericola dumerili*) are generally used as the material for raw fish slices. Chen and Chai (48) examined the sanitation quality of 100 raw sliced fish samples from nine supermarkets and retail stalls. They found that the sanitation quality of raw fish slices in supermarkets is better than that in retail stalls. Based on the levels of aerobic plate count (APC), the nine sampled locations could be separated into two groups (I and II). The average of APC, total coliforms (TC), and volatile base nitrogen (VBN) of raw fish slices in groups I and II are  $2.6 \times 10^5$  and  $3.5 \times 10^6$  CFU/g, 24 and 90 MPN/g, and 10.6 and 11.2 mg/100 g, respectively. They suggested that acceptable limits of sanitation levels of APC, TC, and VBN in raw fish slices were  $5 \times 10^5$  CFU/g, 50 MPN/g, and 15 mg/100 g, respectively. They found that the cloth used for cleaning cutting blocks, knives, and fingers are the sources of recontamination of raw fish slices. In Taiwan and Japan, the slices are conventionally eaten with wasabi paste both for flavor and for the purpose of reducing the microbial load on the slices. However, wasabi can only slightly inhibit the growth of some bacterial strains (49), besides inducing mutation of *Salmonella* spp. (50).

Under the National Shellfish Sanitation Program (NSSP) of the United States, all waters where shellfish are harvested must meet certain standards. Growing waters are classified as (a) approved zone—shellfish may be harvested and sold for human consumption, and the total coliform count in the water should not exceed 70 MPN/ 100 mL; (b) conditionally approved zone—basically clean but known to suffer from predictable periods of contamination, when they are closed; (c) restricted zone—suffering from a certain degree of pollution, but shellfish may be taken from these areas and relayed or depurated in a clean area so that they are safe to market; conditionally restricted zone—as with conditionally approved zone, this covers foreseeable fluctuations in water quality; and (d) prohibited zone—permanently closed to shell fishing either because they are too heavily polluted with sewage or with marine biotoxins or because they have not been surveyed (51).

Fecal coliforms are generally used as indicators for shellfish and its growing water quality. There are two microbiological guidelines applied to fish after harvest. At the wholesale market level, bivalves should have a standard plate count (35°C) of less than  $5.0 \times 10^5/g$  and an MPN of fecal coliforms of less than  $230/100\,g$  (14).

#### 3. Food Safety of Refrigerated and Frozen Seafood

Keeping seafood at low temperature is a common practice to reduce quality degradation. Exposure to low temperature can lead to the leakage of cellular materials (52–54) and degradation of RNA (55, 56), resulting in the injury and death of microorganisms (57). However, injury to the microorganisms by freezing can be repaired to a certain extent during thawing (58). Refrigeration at 5°C stops the growth of the mesophiles, and when the temperature is further lowered, psychrophiles and psychrotrophiles are eliminated. Nonspore-forming gram-negative *Pseudomonas* spp. are cold sensitive, while grampositive *Micrococcus*, *Lactobacillus*, and *Streptococcus* are more resistant (5).

Frozen seafood should to be stored at or below  $-18^{\circ}$ C. During frozen storage, the number of inactive cells formed depends on the storage time. In addition, pretreatment (e.g., bleeding, ice glazing) can affect the quality of seafood during frozen storage. In frozen peeled shrimp, for instance, decomposition can be exacerbated by deteriorated raw materials, inadequate processing conditions, and delayed peeling at room temperature without adequate icing.

Certain pathogenic microorganisms are resistant to freezing temperature. Some V. parahaemolyticus cells inoculated into oysters, sole fillets, and crabmeat can persist at -15 or  $-30^{\circ}$ C with a higher survival ability at  $-30^{\circ}$ C, although there is a sharp reduction in viability during freezing (59). L. monocytogenes has its higher tendency to injury and death at  $-18^{\circ}$ C rather than  $-198^{\circ}$ C (60).

Freezing will bring about a general reduction of the bacterial populations in seafood. This is true for pathogens as well as for psychrophilic spoilage organisms. Generally, gram-negative pathogens such as *Salmonella* and other Enterobacteriaceae are sensitive to freezing injury, and there is also some mortality on mesophilic vibrios. Spores are unaffected by freezing, and vegetative cells of gram-positive bacteria including *Staphylococcus* and *Listeria* usually survive well. During storage of frozen seafood, there is a continued die-off of vegetative bacteria at rates corresponding to the specific species sensitivity and the temperature regime in the storage chamber.

Survival of bacteria in seafood during frozen storage has a real importance for infective organisms such as *Salmonella*, *Shigella*, *Listeria*, *V. cholerae*, and other *Vibrio* spp. since these may be transmitted without further growth and infectivity is dose-related. *Listeria* and *Staphylococcus* survival is significant in terms of frozen seafood in international commerce because of the microorganisms' regulatory significance. Most reports suggest that *V. cholerae* tends to be reduced to very low levels after about 3–6 weeks of storage, but *V. parahaemolyticus* can persist for several months (61)

#### II. FROZEN SEAFOOD HACCP

Though the seafood industry is among the world's oldest industries, it has responded to the challenge of producing a safe product, in addition to being encompassed by other complicated global issues including resource availability, harvesting, and global trade. During the last decade, the United States, the European Economic Community, and other countries have been guided by and later adopted the HACCP approach, formalizing and consolidating their seafood safety control programs.

HACCP is an industry-driven concept that provides a preventive system for hazard control that has been internationally recognized as the most effective tool to secure food safety with current technology. It is based upon a logical, scientific, and systematic

approach to define, identify, and control possible hazards within the food production system. These systems offer greater assurance of food safety and quality and are less dependent upon end product testing. Further, they are designed to introduce on-line process controls that may react more rapidly to potentially hazardous situations.

## A. Procedures in Developing the HACCP Plan

The key element of a HACCP-based system is its preventative nature and its exercising of control throughout the manufacturing process at critical steps called critical control points (CCP). Most companies will find that many of the elements required in a HACCP system are already in place and operable in their plants. Thus the HACCP approach simply takes isolated quality control procedures at various points in the process and puts them all together as an effective control system that specifically focuses on product safety.

The procedure to develop a HACCP system consists of a logical sequence of twelve steps encompassing seven principles. Each of these steps will be discussed in detail along with the process flow for preparing battered and breaded frozen fish fingers, which will be used as an example in elucidating the application of HACCP. The five preliminary steps are

- 1. Assemble the HACCP team.
- 2. Describe the product.
- 3. Describe the intended use and the consumers.
- 4. Develop a process flow diagram.
- 5. Verify the diagram in the operation.

In 1997, the National Advisory Committee on Microbiological Criteria for Foods (NACMCF) of United States has developed seven HACCP principles (62), now accepted worldwide. They are

- 1. Conduct a hazard analysis.
- 2. Identify critical control points.
- 3. Establish critical limits for each critical control point.
- 4. Establish monitoring procedures.
- 5. Establish corrective actions.
- 6. Establish recordkeeping procedures.
- 7. Establish verification procedures.

HACCP is sometimes considered as a two-part system (63). The first part covers from assembling the HACCP team to determining the critical limits associated with the identified CCPs, while the second part consists of monitoring to verification.

## 1. The Preliminary Steps for HACCP Development

## **Step 1: Assemble the HACCP Team**

The initial phase for developing and implementing a HACCP plan for any company is to assemble a multidisciplinary HACCP team that consists of representatives from production, sanitation, quality assurance, food microbiology, engineering, and inspection staff. It is extremely important to get full commitment from management at all levels to the HACCP initiative. Without a firm commitment, the HACCP plan may be more difficult to implement. The HACCP team needs to be aware of the product/process, any food safety programs currently implemented, food safety hazards of concern, and the

seven principles of HACCP. It is also recommended that outside experts who are knowledgeable in the food process or in the areas of food microbiology and microbial pathogens as well as chemical and physical hazards be included in the team or closely associated with the development or verifying the completeness of the HACCP program. However, a plan that is developed solely by outside consultants may lack support by the plant personnel.

Once the HACCP team has been selected and trained, one member of the team must be selected as the team leader. This individual must have managerial and communication skills, be familiar with the plant processes, and preferably be in some position of authority in the company. The team's goal and each member's functions and responsibilities in reaching that goal must be clearly defined.

## **Step 2: Describe the Product**

In order to assist in the identification of possible hazards, the HACCP team must do a complete description of each food product. This should include the raw materials, important end product characteristics, formulation or ingredient, package type, shelf life, label instruction(s), form of distribution, and special distribution control. All this information will assist in the identification of possible hazards that may be inherent in the ingredients or packaging materials, acquired during processing, or generated during storage and distribution.

## Step 3: Identify the Intended Use and Consumers

Identifying the intended use and consumer is to identify how the product will be used by the normal end users or consumers, for examples, to be eaten without further cooking, or to be fully cooked before consumption. Further, the HACCP team must identify any particular segment of the intended population that is at increased risk, such as infants or the elderly. In some cases, the intended user may be another processor, who will further process the product. Table 3 shows the product description and the intended use and consumer of battered and breaded pollock fish fingers.

 Table 3
 HACCP Plan: Product Description and the Intended Use and Consumer

Product description					
1. Product name(s)	Battered and breaded frozen pollock fish finger				
2. Important product characteristics	Prefried, not fully cooked, IQF quick frozen				
3. How it is to be used	Fully cooked before serving				
4. Packaging	Vacuum packed plastic bags, assorted sizes				
5. Shelf life	One year frozen				
6. Where it will be sold	Retail and wholesale with frozen shelf				
7. Labeling instructions	Not fully cooked, keep frozen, heated before serving				
8. Special distribution requirements	Vehicles with freezer capabilities				
Intended use and consumer					
1. Intended use	Cooked by consumer before serving				
2. Intended consumer	General population				

## Step 4: Develop a Process Flow Diagram

The process flow diagram will identify the important process steps used in the production of the specific product being reviewed. The flow diagram is designed to provide a complete description of all of the steps involved in the operations from raw material receiving through to shipment of finished products. This is to provide a clear, simple description of the steps involved in the processing of the fishery product and its associated ingredients.

It should be noted that in some countries, food regulations also require that a plant schematic should also be developed to show product flow and employee traffic patterns within the plant for the specific product. Figure 2 shows an example of a flow diagram for battered and breaded pollock fish fingers.

## Step 5: Verify the Diagram in the Operation

Once the process flow diagram and/or plant schematic has been prepared, it must be verified by an on-site inspection for accuracy and completeness by the HACCP team. This will ensure that all major steps have been identified and further validate the movement of product and employees in the food plant.

## 2. The Seven HACCP Principles

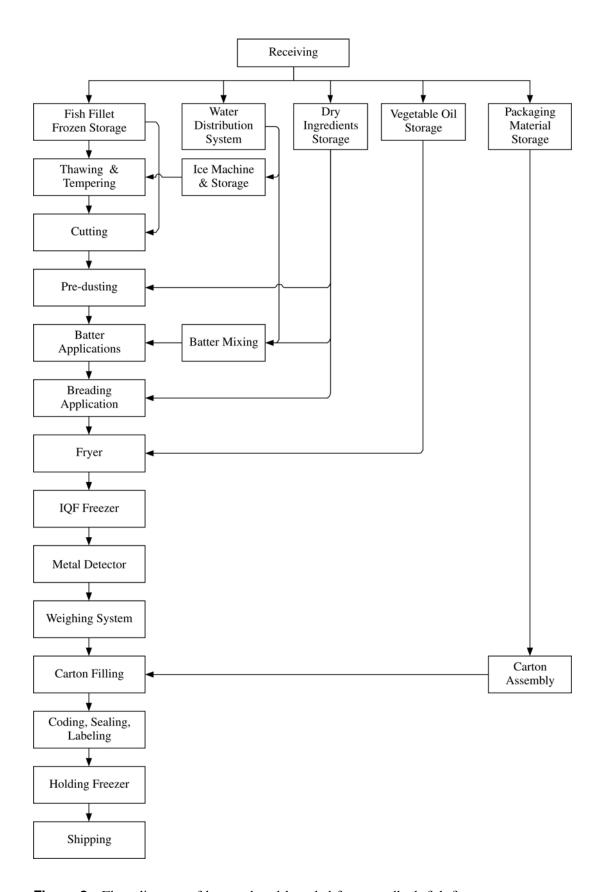
## Principle 1: Conduct a Hazard Analysis

The first step in the development of a HACCP plan is to identify those hazards associated with the product. This step accomplishes three purposes: identifying all possible potential hazards, selecting significant hazards based on a risk-management approach, and developing preventive measures for every identified significant hazard. Table 4 shows an example of a hazard analysis worksheet for battered and breaded pollock fish fingers.

a. Identifying Potential Hazards. A hazard may be biological, chemical, or physical in nature, and its existence in a product can lead to harmful results when consumed. Thus a wrong hazard analysis inevitably leads to the development of an inadequate HACCP plan. Before starting hazard identification and analysis, a brief literature search should be carried out. This routine will provide the HACCP team with an updated and scientific review of general as well as specific information related to the identification and control of food safety hazards for the product examined. The information can be obtained from scientific research or review papers, official epidemiological reports, reference texts, reference databases built and maintained within the company, and the company's complaints files.

Numerous factors, such as ingredients, processing, distribution, and the intended use of the product have to be considered during hazard analysis. The significant hazards associated with each step in the flow diagram should be listed along with preventative measures designed to control the hazards. All the information will be tabulated in a Hazard Analysis Worksheet and combined with Principle 2 to determine the CCPs.

Seafood-borne biological hazards include bacterial, viral, and parasitic organisms. These organisms are commonly associated with raw fishery products entering the processing plant. Fish flesh is an excellent substrate for the growth of most heterotrophic bacteria. Pathogenic microorganisms associated with seafood are categorized according to whether they originate in the living animal, in polluted water, or from postcapture/harvest contamination, as well as additional pathogens may be introduced as the result of ingredients or batter and breading. The only pathogens for humans indigenous to the



**Figure 2** Flow diagram of battered and breaded frozen pollock fish fingers.

 Table 4
 Hazard Analysis Worksheet of Battered and Breaded Frozen Pollock Fish Fingers

(1)	(2)	(3) Are any potential	(4)	(5)	(6) Is this step a critical control point? (Yes/No)	
Ingredient/processing step	Identify potential hazards introduced, controlled or enhanced at this step (1)	food-safety hazards significant? (Yes/No)	Justify your decisions for column 3.	What preventive measures can be applied to prevent significant hazards?		
Fish fillet receiving	Biological: contaminated with pathogens	Yes	Raw fish is known source of pathogens.  Proper temperature control to reduce pathogen growth; subsequent heating step should help to reduce their level.		No	
	Parasites	No	Parasites are killed during extended frozen storage.			
	Chemical: sanitizers, clearners, etc.	No	Unlikely to occur; suppliers' SSOP should control.			
	Physical: foreign matter	No	Low risk, unlikely to occur.			
Fish fillet frozen storage	Biological: pathogen growth during storage Chemical: None Physical: None	No	Unlikely to occur. Controlled by prerequisite programs: storage.			
Thawing & tempering	Biological: None during operation Chemical: None Physical: None	No	Unlikely to occur. Controlled by prerequisite programs: storage.			
Cutting	Biological: pathogen growth during operation	No	Unlikely to occur. Controlled by SSOP.			
	Chemical: sanitizers, clearners, etc.	No	Unlikely to occur. Controlled by SSOP.			
	Physical: metal fragments	Yes	Machine parts may fall off and mix with meat mixture	All packaged products are checked by a metal detector.	No	
Receiving and storing of	Biological: spores	No	B, C, and P: unlikely to occur, suppliers'			
nonfish ingredients and packaging materials	Chemical: contaminated with toxins	No	letter of guarantee, certificate of analysis.			
	Physical: foreign matter	No				
Weighing and mixing of nonfish ingredients	Biological: pathogen growth during operation	No	Unlikely to occur. Controlled by SSOP.			
	Chemical: None	No	Unlikely to occur. Controlled by SSOP.			
	Physical: fragment inclusions	Yes	Foreign inclusions may be mixed with ingredients.	All packaged products are checked by a metal detector.	No	
Predusting	Biological: pathogens grow during operation Chemical: None	No	Unlikely to occur during such a short duration.			
	Physical: metal fragments	Yes	Machine parts may fall off and mix with meat mixture	All packaged products are checked by a metal detector.	No	
					(Continued)	

 Table 4 (Continued)

(1)	(2)	(3) Are any potential	(4)	(5)	(6) Is this step a critical control point? (Yes/No)	
Ingredient/processing step	Identify potential hazards introduced, controlled or enhanced at this step (1)	food-safety hazards significant? (Yes/No)	Justify your decisions for column 3.	What preventive measures can be applied to prevent significant hazards?		
Batter application	Biological: pathogens grow during operation	Yes	Staphylococcus aureus may grow and produce toxin during batter mix recirculation.	Time/temperature control to reduce potential microbial growth.	Yes	
	Chemical: None					
	Physical: metal fragments	Yes	Machine parts may fall off and mix with meat mixture.	All packaged products are checked by a metal detector.	No	
Breading application	Biological: pathogens grow during operation Chemical: None	No	Unlikely to occur during such a short duration.	ŕ		
	Physical: metal fragments	Yes	Machine parts may fall off and mix with meat mixture.	All packaged products are checked by a metal detector.	No	
Fryer	Biological: None Chemical: None Physical: None			, and the second		
IQF freezer	Biological: None Chemical: None Physical: None					
Weighing	Biological: None Chemical: None					
Packaging	Physical: None Biological: None Chemical: None					
Metal detector	Physical: None Biological: None Chemical: None					
	Physical: foreign objects and metal inclusions	Yes	Any harmful physical foreign objects in the product may present threat to consumers.	All packaged products are checked by a metal detector.	Yes	
Frozen storage	Biological: pathogen growth during storage Chemical: None Physical: None	No	Unlikely to occur. Controlled by prerequisite programs: storage.			
Shipping	Biological: pathogen growth during storage	No	Unlikely to occur. Controlled by prerequisite programs: transportation.			
	Chemical: None Physical: None					

marine environment or marine animals are *C. botulinum* and various *Vibrio* spp. (64). Other human pathogens may contaminate fish taken from waters subject to pollution from warm-blooded animals, and this is particularly important for mollusks such as oysters, mussels, and clams, which acquire nutrients by filtering large volumes of water.

Animal parasites live in or on other animals from which they obtain at least some of their vital requirements, particularly nutrients. The species most frequently found are worms, or nematodes, among which two kinds predominate, the cod worm (*Phocanema decipiens*) and the herring worm (*Anisakis simplex*). There have been cases of human illness caused by the ingestion of live *Phocanema* or *Anisakis* larvae in countries where raw or lightly cured fish is commonly consumed (65).

Chemical contaminants may be naturally occurring or may be added during the processing of food. Harmful chemicals at very high levels have been associated with acute cases of food-borne illnesses and can be responsible for chronic illness at lower levels. Types of chemical hazards include naturally occurring chemicals such as marine toxins, and chemicals intentionally or unintentionally added to the products. The composition of free amino acids in fish flesh can be harmful to human health through formation of biogenic amines. Fish tissues contain high levels of free nonprotein nitrogen (NPN) compounds such as trimethylamine oxide (TMO), which is typically reduced to TMA by spoilage bacteria, thus producing the characteristic "fishy" smell of spoiled fish (8). Another good example is histidine, which is present in high concentrations in certain fish species and is converted to histamine by enzymes. It is important to note that though freezing halts the production of histidine decarboxylase by bacteria, the enzyme continues to be active. This can result in significant elevation of histamine, even above the harmful level of 50 ppm, during long-term frozen storage.

The principal seafood-associated intoxications having a microbiological origin include paralytic shellfish poisoning (PSP), diarrhetic shellfish poisoning (DSP), neurotoxic shellfish poisoning (NSP), amnesic shellfish poisoning (ASP, also known as domoic acid poisoning), ciguatera, and scombroid (biogenic amine) fish poisoning (66).

Illness and injury can also result from physical hazards, such as hard foreign objects in the product. These hazards can be introduced into products from contamination and/or poor hygienic procedures at many points in the food chain from harvest to consumer. For frozen seafood, the most commonly found physical hazards are bone fragments, stones, metal fragments, or plastic peeled from packaging material.

b. Selecting Significant Hazards. The analysis of hazards requires the assessment of two factors with respect to any identified hazard, i.e., the likelihood that the hazard will occur, and the severity of it if it does occur. Water temperature has been proven to be a major factor for the presence of mesophilic vibrios in marine animals; they grow rapidly at temperatures between 20 and 40°C, reflected in the high content of vibrios isolated from molluscan shellfish when water temperatures rise to 30°C, and their virtual absence from mollusks taken from cold waters (47). Therefore when conducting hazard analysis for this pathogen, the history of the water temperature of the harvesting area should also be considered in determining whether the biological hazard is significant.

Seafood toxins have been responsible for nearly two-thirds of all seafood-borne outbreaks of illness in the U.S., with the majority of cases being associated with consumption of finfish, where epidemiological data also suggest that a similar situation exists globally (47). The two most common intoxications have been ciguatera and scombroid poisonings. Once they are present in fishery raw materials, they can hardly be detected organoleptically or by routine microbiological analysis. More importantly, all seafood toxins are heat resistant and are not destroyed by cooking (8).

c. Preventive Measures for Significant Hazards. There are a number of strategies for the control of pathogens in fish and fishery products. These include managing the amount of time that food is exposed to temperatures that are favorable for pathogen growth and toxin production; killing pathogens by cooking, pasteurizing, or retorting; controlling the amount of moisture that is available for pathogen growth, water activity, in the product; controlling the amount of salt or preservatives, such as sodium nitrite, in the product; controlling the level of acidity, pH, in the product (66). Whenever possible, the source of the finfish should be inspected organoleptically for obvious signs of spoilage.

There are a number of potential public health concerns associated with fresh finfish, but in most instances these are controlled by the same means used to prevent spoilage. Temperature is the single most important factor affecting the rate of fish deterioration and the multiplication of microorganisms. For species prone to scombroid toxin production, time and temperature control may be the most effective method in controlling food safety. It is therefore essential that fresh fish, fillets, and other shellfish and their products that are to be chilled should be held at a temperature as close as possible to 0°C.

Maintaining refrigerated storage temperatures as low as possible ( $<2^{\circ}$ C) helps prevent or delay the growth of psychrotrophic pathogens (*L. monocytogenes*, *Yersinia enterocolitica*, nonproteolytic *C. botulinum*, etc.) (67).

In addition to the controls associated with raw fish that have already been discussed, frozen seafood has two other factors that must be considered, rate of freezing, and temperature control during frozen storage. Temperature control is the principal means to stop microbial activity in frozen seafood. Freezing should be as rapid as possible, and once frozen the product should be held at or below  $-18^{\circ}$ C. The primary means of assessing the effectiveness of these controls is through monitoring the product and the storage environment. The thawing of the product can also have a strong influence on the microbiological quality and safety of the product. Thawing should be as rapid as possible and should avoid having the exterior surface of the product exposed to abusive temperatures while waiting for the center to thaw.

Ciguatera and algal intoxications are best handled through the control of finfish sources; and harvesting from warning alert areas or during an algal bloom should be avoided. This is currently achieved through periodic assessment of fishing grounds for toxic algae and the avoidance of certain large fish from high-risk areas.

Nematode larvae are resistant to salting; immersion in  $80^{\circ}$  brine (21% salt by weight) for 28 days will not kill all such larvae. When there is doubt whether parasites will survive a process, it is safest to use frozen fish. Finfish intended to be consumed raw should be frozen for a sufficient amount of time to assure inactivation of the organisms. Freezing of fish at  $-20^{\circ}$ C for 7 days or  $-35^{\circ}$ C for 15 hours kills all parasites (66). This procedure is recommended for raw products to be eaten as sashimi. Freezing does not affect marine toxins accumulated in the living animal or bacterial toxins produced during inappropriate handling before freezing.

A way to reduce the numbers of parasites reaching the consumer is to inspect the fish. Parasites embedded deep in the flesh are not immediately obvious, but some can be detected by shining a bright light through the fillet: this is called candling. In commercial practice, candling and trimming away the belly flaps of fish are effective in reducing the numbers of parasites (65). However, they do not completely eliminate the hazard, nor do they minimize it to an acceptable level.

## **Principle 2: Identify Critical Control Points**

A CCP is a point, step, or procedure at which control can be applied and a food safety hazard prevented, eliminated, or reduced to acceptable levels. The determination of a CCP is based on the assessment of the severity and likely occurrence of hazards. A separate CCP does not have to be designed for each hazard. However, actions must be taken to ensure that all identified hazards are excluded. Examples of CCPs may include receiving, cooking, chilling, and product formulation control. Likewise, refrigeration or the adjustment of a product's pH to a level required, preventing hazardous microorganisms from multiplying or toxins from forming, are also CCPs.

CCPs can be determined with the aid of HACCP decision tree that was first developed by Codex Alimentarius working groups in 1991. Since then it has been modified based on suggestions made by researchers, official inspectors, and industry. Its flow of questions and usage are readily available elsewhere.

Many points in seafood processing may be considered control points, but very few are actually critical control points. A control point is any point, step, or procedure at which biological, physical, or chemical factors can be controlled. Concerns that do not impact food safety may be addressed at control points; however, since these control points do not relate to food safety, they are not included in the HACCP plan.

## Principle 3: Establish Critical Limits for Each Critical Control Point

In order to safeguard the safety of a product, a criterion must be met for each preventive measure associated with a CCP. Critical limits can be thought of as boundaries of safety for each CCP that separate acceptability from unacceptability. Typical criteria may be set for preventive measures such as temperature, time, physical dimensions, water activity, pH, and available chlorine. Critical limits may be derived from sources such as regulatory standards and guidelines, consultations with experts, or other scientific data. The processor is responsible for using competent authorities to validate that the critical limits chosen will control the identified hazard.

## **Principle 4: Establish Monitoring Procedures**

Monitoring is a planned sequence of observations or measurements to assess whether a CCP is under control. There are three main purposes for monitoring: it tracks the operation so that a trend toward a loss of control can be recognized and corrective action can be taken to bring the process back into control before a deviation occurs; it indicates when a loss of control and a deviation have actually occurred, and corrective action must be taken; and it provides written documentation for use in the verification of the HACCP plan.

Monitoring procedures are preferably rapid type tests, visual examination and documentation, or any appropriate routines because they relate to on-line process and there is not sufficient time for lengthy analytical testing. Continuous monitoring is always advantageous when feasible, and with modern technologies, it is possible with many types of physical and chemical methods. Monitoring, corrective actions, and record keeping are usually considered the "active components" of an HACCP system.

The designated monitoring frequency must be sufficient to demonstrate that the hazard is under control. Responsibility for monitoring is clearly identified, and individuals monitoring the CCPs must be trained in the testing procedure and must be fully aware of the purpose and importance of monitoring. All monitoring equipment used by the seafood

processors for measuring critical limits must be carefully calibrated for accuracy. Records of calibrations must be maintained as a part of the HACCP plan documentation.

#### **Principle 5: Establish Corrective Actions**

Although the HACCP system is intended to prevent deviations from occurring, perfection is rarely, if ever, achievable. Corrective actions are a predetermined and documented set of actions that are implemented when a deviation occurs. Corrective actions include the plan in place to determine the disposition of any products produced when a deviation was occurring, correct the cause of the deviation and ensure that the critical control point is under control, and maintain records of corrective actions.

The diverse nature of possible deviations usually means that more than one corrective action may be required at each CCP. It must correct the cause of the problem and must control the actual or potential hazard resulting from the deviation. Product control includes proper identification and handling of the affected lots. Corrective action procedures must be documented in the HACCP plan. Since corrective actions are prescribed and formalized, employees responsible for CCP monitoring understand and are able to perform the appropriate corrective actions in the event that control failure occurs. Table 5 shows an example of the HACCP plan form of battered and breaded pollock fish fingers.

#### **Principle 6: Establish Record-Keeping Procedures**

This principle requires the preparation and maintenance of an up-to-date written HACCP plan by the food processor. The plan must detail the hazards of each individual or categorical product covered by the plan and also clearly identify the CCPs and critical limits for each CCP. CCP monitoring and record-keeping procedures must be shown in the establishment's HACCP plan.

The HACCP records are done at each CCP, and they contain the information required to ensure that the HACCP plan is followed. The record-keeping associated with HACCP procedures ultimately makes the system work.

A record may be in any form. Processing charts, written records, and computerized records are all valid and show the historical record of the process, the monitoring, the deviations, and the corrective actions that occurred at the identified CCPs. The records are important evidence that a supervisor or inspector must have to ensure that the establishment is following the agreed-upon HACCP plan.

## **Principle 7: Establish Verification Procedures**

Verification activities are methods, procedures, and tests that differ from monitoring activities in that the results are not intended to make decisions on the acceptability of lots of product; rather, they are used to determine if the HACCP plan for the product or process is valid and operating properly.

It has been categorized into four phases of activities. The first phase of the process includes scientific or technical verification that critical limits at CCPs are satisfactory. The second phase of verification involves a frequent reviewing of CCP records that ensure that the facility's HACCP plan is functioning effectively. The third phase consists of periodic revalidations, independent of audits or other verification procedures that are carried out to ensure the accuracy of the HACCP plan. The fourth phase of verification deals with the

 Table 5
 HACCP Plan Form for Battered and Breaded Frozen Pollock Fish Fingers

Critical control point (CCP)	Significant hazard(s)	Critical limits for each preventive  Measure	Monitoring						
			What	How	Frequency	Who	Corrective action(s)	Records	Verification
Batter application	S. aureus growth and toxin formation	Hydrated batter mix temperature not to exceed 10°C for more than 12 h, nor 18°C for more than 3 h, cumulative	Hydrated batter mix temperature	Recorder thermometer	Continuous with visual check every 2h	Production employee	Adjust hydrated batter mix refrigeration equipment Destroy hydrated batter mix and any product produced during deviant period	Recorder thermometer chart	Check accuracy of recorder thermometer once per day. Review monitoring, corrective action every week. Calibrate thermometer every year.
Metal detector	Metal inclusion	No detectable metal fragments in finished product	Presence of detectable metal fragments in finished product	Metal detector	Every finished product package, with operation check before start-up	Production employee	Destroy any product rejected by metal detector. Identify source of metal found in product and fix damaged equipment. If product is processed without metal detection hold for metal detection.	Metal detector operation log	Test metal detector with three test units before production each day. Review monitoring, corrective action every week.

regulatory agency's responsibility and inspection to ensure that the establishment's HACCP system is functioning satisfactorily.

Verification activities are generally involved and may include analytical testing. Microbial analysis of frozen seafood is largely limited to providing information on the quality of the product before it was frozen, and the extent that microflora have changed as a result of frozen storage. *Staphylococcus* and *Listeria* are relatively resistant to the effects of freezing and may provide some indication of the degree of human contact with the product and the extent of contamination in the processing environment.

# III. RECOMMENDATIONS FOR FISH CATCHING AND HANDLING BEFORE IN-PLANT PROCESSING

To reduce the deterioration of seafood freshness, quality-keeping operations should be carried out immediately after catching or harvesting. In the final section of this chapter, we will discuss the Torry Advisory Notes for the catches and the practical operations aboard fishing vessels, on fish markets, and on land transport.

## A. On Board Fishing Vessels

This section attempts to highlight the basic requirements for cleanability and for minimizing damage, contamination, and decomposition, which all vessels should have to the extent possible, in order to ensure hygienic, high-quality handling of fresh fish intended for further processing or freezing. Captains of trawling vessels should determine the times to lift nets during trawling. When fish are netted, they will struggle and then die. The immune systems of the organisms will lose their activities after death. The flesh thus will be degraded by intrinsic enzymes and microorganisms in the intestine or from the seawater and sediments. For keeping good quality in the catches, trawling vessels operated in warm water (e.g., 28°C) can be arranged to lift their nets within at least 4 hours each time. In cold water (e.g., 10°C), the time can be longer (e.g., 24 hours). Periods of higher temperatures should be minimized and should not exceed 2 h. On fishing vessels on which primary processing operations such as heading, gutting, and filleting are conducted, there should be sufficient cold storage space to chill and hold fresh whole product between catching and processing. For instance, with large fish such as tuna and swordfish, evisceration can be carried out as soon as possible after harvest. Every piece of gut and liver should be removed. Both napes on round fish are cut, where permissible, to allow the belly cavity to be properly washed. Guts should not be dropped on top of other fish. They should be put in a basket or thrown away properly. The sorted and/or above-treated fish and shellfish are then placed in fish boxes or baskets that are covered with crushed ice or stored in freezing pens. Refrigerated brine-holding tanks should be emptied and refilled with clean seawater or brine between fishing trips to avoid excessive buildup of spoilage bacteria. Wash water and ice should be clean and free of contamination. In no case should previously used ice be reused directly to cool fresh fish. Birds, insects, and other animals present a serious contamination hazard and must be controlled along with proper offal disposal and frequent sanitary cleanup. All facilities and equipment should be cleaned and sanitized on a routine basis.

Chilling a fish catch by crushed ice is proper only on short (within 15 days) fishing operations. By this method, the fish is placed into crushed ice as quickly as possible. Plenty of ice (one part of fish to more than two parts of ice) should be used to prepare a good

layer below the fish, more ice between them, and another layer on top. For a long fishing trip, extended to several months, a proper freezing or cold storage operation at sea is required to keep good quality in the catches. Freezing at sea has been well developed for most fishing vessels. Fish should be precooled to  $0-4^{\circ}$ C prior to freezing at  $-30^{\circ}$ C or lower. It is important to reduce whole fish body temperature to  $-18^{\circ}$ C or lower as soon as possible.

Decks should be cleaned prior to loading the first haul that comes aboard. In fish handling areas, surfaces should have a minimum of sharp corners and projections. In boxing and shelving fish storage areas, the design should preclude excessive pressure being exerted on the fish. Chutes and conveyors should be designed to prevent physical damage caused by long drops or crushing. Sea gulls fly over the vessels and excrete their feces that may occasionally drop down to the decks. Feces thus are the primary contaminants of intestinal bacteria on seafood. Disinfection of the decks can be carried out using chlorinated seawater or other disinfectants after cleaning using detergents.

Where appropriate, adequate facilities should be provided for the handling and washing of fish and should have an adequate supply of cold potable water or clean seawater for that purpose. After loading on the decks, the catch can be hosed with seawater or any clean water. Objectionable substances, which could include bilge water, smoke, fuel oil, grease, drainage and other solid or semisolid wastes, should not contaminate the fish.

#### B. In Fish Markets

In some areas of the world, fishing vessels unload their fish catches directly to the processing plants. Most vessels, however, land their catches in fish markets in fishing ports. During unloading and landing, contamination of fishery products must be avoided. It must in particular be ensured that unloading and landing operations proceed rapidly. Fishery products are placed without unnecessary delay in a protected environment at the temperature required on the basis of the nature of the product and, where necessary, in ice in transport, storage, or market facilities. Equipment and handling practices that cause unnecessary damage to the edible parts of fishery products are not allowed.

The markets should furnish a sanitary environment to prevent the catches from contamination and freshness reduction. Rapid auction is required to keep good seafood quality. Standing on frozen blocks of sea-frozen fish is not permitted during auction or any time, since fish is food. If it can be avoided to unload cargo on the floor of the market, the blocks should be transferred directly to a cold store by conveyor belt. Otherwise the blocks should be stacked quickly and carefully on wooden or metal bins or pallets and rapidly moved to the cold store.

After landing or, where appropriate, after first sale, fishery products must be transported without delay to their destinations. However, in markets where fishery products may be stored before being displayed for sale, or after being sold, and pending transport to their destinations, there must be sufficiently large cold rooms available so that fishery products can be stored at a temperature approaching that of melting ice.

## C. Land Transport

The storage life of frozen fish depends upon storage temperature. Any increase in temperature, even for a very short time, has a bad effect on the quality of the product. Poor handling practices can lead to damage in fresh fish that can accelerate the rate of

decomposition and increase unnecessary postharvest losses. Handling damage can be minimized by handling and conveying with care, particularly during transfer and sorting, in order to avoid physical damage such as puncture and mutilation. Where boxes are used for the storage of fish, they should not be overfilled or stacked too deeply.

One of the most difficult problems in the distribution of frozen fish is to move it from one store to another, or from cold store to display cabinet at the retailer's shop, without too great a rise in temperature during the journey. Containers are usually employed to deliver frozen fish for a long road journey. These containers should be designed to deliver the last of the load at a temperature not higher than  $-18^{\circ}$ C. It is recommended to reduce loading and unloading time, and to protect the cargo at these times. Insulation must not be compromised. A good-quality material should be used and applied sufficiently thickly.

During transport, frozen fishery products, with the exception of frozen fish in brine intended for the manufacture of canned foods, must be kept at an even temperature of  $-18^{\circ}$ C or less in all parts of the product, allowing for the possibility of brief upward fluctuations of not more than  $3^{\circ}$ C. Products may not be stored or transported with other products that may contaminate them or affect their hygiene, unless they are packaged in such a way as to provide satisfactory protection.

Vehicles used for the transport of fishery products must be constructed and equipped so that the temperatures can be maintained throughout the period of transport. If ice is used to chill the products, adequate drainage must be provided in order to ensure that water from melted ice does not stay in contact with the products. The inside surfaces of the means of transport must be finished in such a way that they do not adversely affect the fishery products. They must be smooth and easy to clean and disinfect. Means of transport used for fishery products may not be used for transporting other products likely to impair or contaminate fishery products, except where the fishery products can be guaranteed uncontaminated as a result of such transport being thoroughly cleaned and disinfected.

In conclusion, since there is no substitute for good raw material and most seafood tends to decompose rapidly, the final product will never be any better than the raw material. Storage of seafood at low temperatures can prolong the shelf life of products. Freezing cannot sterilize microorganisms in seafood. Frozen storage can only retard the degradation of seafood. Proper care in the harvesting, receiving, processing, holding, and storage of raw materials and products must always be exercised and will provide hazard control to secure frozen seafood safety.

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# 21

## **Frozen Seafood: Product Descriptions**

## **Peggy Stanfield**

Dietetic Resources, Twin Falls, Idaho, U.S.A.

This book is not the proper forum to discuss the manufacture of every frozen seafood product available in the market. However, regulatory agencies such as the National Marine Fisheries Service (NMFS) have issued some minimal criteria for several frozen seafood and seafood products: what they are, what types and styles are available, and so on. The information in this chapter describes each available frozen seafood product and has been modified from the product grades issued by the NMFS. A product grade is established to achieve two objectives: assure product safety and minimize economic fraud.

#### I. FROZEN HEADLESS DRESSED WHITING

## A. Description of the Product

The product described in this part consists of clean, wholesome whiting (silver hake) *Merluccius bilineraris*, *Merluccius albidus* completely and cleanly headed and adequately eviscerated. The fish are packaged and frozen in accordance with good commercial practice and are maintained at temperatures necessary for the preservation of the product.

#### B. Grades of Frozen Headless Dressed Whiting

U.S. Grade A is the quality of frozen headless dressed whiting that possess a good flavor and odor. U.S. Grade B is the quality of frozen headless dressed whiting that possess at least reasonably good flavor and odor. Substandard or Utility is the quality of frozen headless dressed whiting that otherwise fail to meet the requirements of U.S. Grade B.

#### C. Determination of the Grade

Good flavor and odor (essential requirements for a U.S. Grade A product) means that the cooked product has the typical flavor and odor of the species and is free from rancidity, bitterness, staleness, and off-flavors and off-odors of any kind.

Reasonably good flavor and odor (minimum requirements of a U.S. Grade B product) means that the cooked product is lacking in good flavor and odor but is free from objectionable off-flavors and off-odors of any kind.

Arrangement of product refers to the packing of the product in a symmetrical manner, bellies or backs all facing in the same direction, fish neatly dovetailed.

Condition of the packaging material refers to the condition of the cardboard or other packaging material of the primary container.

If the fish is allowed to stand after packing and prior to freezing, moisture from the fish will soak into the packaging material and cause deterioration of that material.

Dehydration refers to the presence of dehydrated (water-removed) tissue on the exposed surfaces of the whiting. Slight dehydration is surface dehydration that is not color masking. Deep dehydration is color masking and cannot be removed by scraping with a fingernail.

Minimum size refers to the size of the individual fish in the sample. Fish 2 ounces or over are considered acceptable. Smaller fish cannot be cooked uniformly with acceptable size fish.

Heading refers to the condition of the fish after they have been headed. The fish should be cleanly headed behind the gills and pectoral fins. No gills, gill bones, or pectoral fins should remain after the fish have been headed.

Evisceration refers to the cleaning of the belly cavities of the fish. All spawn, viscera, and belly strings should be removed.

Scaling refers to the satisfactory removal of scales from the fish.

Color of the cut surfaces refers to the color of the cut surfaces of the fish after heading and other processing.

Bruises and broken or split skin refers to bruises over one-half square inch in area and splits or breaks in the skin more than one-half inch in length that are not part of the processing.

Texture defects refers to the absence of normal textural properties of the cooked fish flesh, which are tenderness, firmness, and moistness without excess water. Texture defects are dryness, softness, toughness, and rubberyness.

#### II. FROZEN HALIBUT STEAKS

#### A. Product Description

Frozen halibut steaks are clean, wholesome units of frozen raw fish flesh with normally associated skin and bone and are 2 ounces or more in weight. Each steak has two parallel surfaces and is derived from whole or subdivided halibut slices of uniform thickness, which result from sawing or cutting perpendicular to the axial length, or backbone, of a whole halibut. The steaks are prepared from either frozen or unfrozen halibut (*Hippoglossus* spp.) and are processed and frozen in accordance with good commercial practice and are maintained at temperatures necessary for the preservation of the product.

## B. Styles of Frozen Halibut Steaks

#### 1. Style I—Random Weight Pack

The individual steaks are of random weight and neither the weight nor the range of weights is specified.

## 2. Style II—Uniform Weight or Portion Pack

All steaks in the package or in the lot are of a specified weight or range of weights.

#### C. Recommended Dimensions

- 1. The recommended dimensions of frozen halibut steaks are not incorporated in the grades of the finished product, since dimensions as such are not factors of quality for the purpose of these grades. However, the degree of uniformity of thickness among units of the finished product is rated, since it is a factor affecting the quality and utility of the product.
- 2. It is recommended that the thickness (smallest dimension) of individually frozen halibut steaks be not less than  $\frac{1}{2}$  inch and not greater than  $\frac{1}{4}$  inches.

Percentage glaze on halibut steak means the percentage by weight of frozen coating adhering to the steak surfaces and includes the frost within the package.

Uniformity of thickness means that the thickness is substantially the same for one or more steaks within a package or sample unit.

#### D. Color Defects

- 1. Discoloration of drip liquor means that the free liquid that drains from the thawed steaks is discolored with blood residue usually from the dorsal aorta of the halibut.
- 2. Discoloration of light meat means that the normal flesh color of the main part of the halibut steak has darkened owing to deteriorative influences.
- 3. Discoloration of the dark meat means that the normal color of the surface fat shows increasing degrees of yellowing due to oxidation.
- 4. Nonuniformity of color refers to noticeable differences in color on a single steak or between adjacent steaks in the same package.
- 5. Dehydration refers to the appearance of a whitish area on the surface of a steak due to the removal of water or drying of the affected area.
- 6. Honeycombing refers to the visible appearance of numerous discrete holes or openings of varying size on the steak surface.
- 7. Workmanship defects refers to appearance defects that were not eliminated during processing and are considered either objectionable or poor commercial practice.
- 8. Texture defect refers to an undesirable increase in toughness and/or dryness, fibrousness, and watery nature of the halibut examined in the cooked state.

#### III. FROZEN SALMON STEAKS

#### A. Product Description

Frozen salmon steaks are clean, wholesome units of frozen raw fish flesh with normally associated skin and bone and are 2.5 ounces or more in weight. Each steak has two parallel surfaces and is derived from whole or subdivided salmon slices of uniform thickness that result from sawing or cutting dressed salmon perpendicularly to the axial length, or backbone. The steaks are prepared from either frozen or unfrozen salmon (*Oncorhynchus* spp.) and are processed and frozen in accordance with good commercial practice and are maintained at temperatures necessary for the preservation of the product. The steaks in an individual package are prepared from only one species of salmon.

## **B.** Species

Frozen salmon steaks covered huiby are prepared from salmon of any of the following species:

Silver or coho (O. kisutch)
Chum or keta (O. keta)
King, chinook, or spring (O. tshawytscha)
Red, sockeye (O. nerka)
Pink (O. gorbuscha)

## C. Styles of Frozen Salmon Steaks

#### 1. Style I—Random Weight Pack

The individual steaks are of random weight, and neither the individual steak weight nor the range of weights is specified. The steaks in the lot represent the random distribution cut from the head to tail of a whole dressed salmon.

## 2. Style II—Random Weight Combination Pack

The individual steaks are of random weight, and neither the individual steak weight nor the range of weights is specified. The steaks in the lot represent a combination of cuts from selected parts of the whole dressed salmon.

#### 3. Style III—Uniform Weight or Portion Pack

All steaks in the package or in the lot are of a specified weight or range of weights.

## D. Recommended Dimensions

It is recommended that the thickness (smallest dimension) of individually frozen salmon steaks be not less than  $\frac{1}{2}$  inch and not greater than  $\frac{1}{2}$  inches.

General appearance defects refer to poor arrangement of steaks, distortion of steaks, wide variation in shape, between steaks greater than normal number of head and/or tail pieces, imbedding of packaging material into fish flesh, inside condition of package, frost deposit, excessive or nonuniform skin glaze, and undesirable level of natural color.

Dehydration refers to the appearance of a whitish area on the surface of a steak due to the evaporation of water or drying of the affected area.

Uniformity of thickness means that the steak thickness is within the allowed manufacturing tolerance between the thickest and thinnest parts of the steaks within a package or sample unit.

Workmanship defects refers to appearance defects that were not eliminated during processing and are considered objectionable or poor commercial practice. They include the following: blood spots, bruises, cleaning (refers to inadequate cleaning of the visceral cavity from blood, viscera, and loose or attached appendages), cutting (refers to irregular, inadequate, unnecessary, or improper cuts and/or trimmings), fins, foreign material (refers to any loose parts, of fish or other than fish origin), collar bone, girdle (refers to bony structure adjacent to fin), loose skin, pugh marks, sawdust, and scales.

#### E. Color Defects

Discoloration of fat portion means that the normal color of the fat shows increasing degrees of yellowing due to oxidation.

Discoloration of lean portion means that the normal surface flesh color has faded or changed due to deteriorative influences.

Nonuniformity of color refers to noticeable differences in surface flesh color on a single steak or between adjacent steaks in the same package or sample unit. It also includes color variation of the visceral cavity and skin watermarking.

Honeycombing refers to the visible appearance on the steak surface of numerous discrete holes or openings of varying size.

Texture defect refers to an undesirable increase in toughness and/or dryness, fibrousness, and watery nature of salmon examined in the cooked state.

#### IV. FROZEN FISH FILLET BLOCKS

## A. Product Description

Frozen fish blocks are rectangularly shaped masses made from a single species of fish flesh. They are made from fillets or fillet pieces that are either skin-on and scaled or skinless. Blocks processed from skin-on fish flesh should be so labeled. The blocks should not contain minced or comminuted fish flesh. The blocks should not be made by restructuring (reworking) pieces of fish blocks into the shape of a fish block.

## 1. Dehydration

This defect refers to loss of moisture from the surface of a fish block during frozen storage. Affected areas have a whitish appearance. Moderate dehydration masks the surface color of the product and affects more than 5% up to and including 15% of the surface area. If more then 15% of the surface area is affected, each additional 15% of surface area affected is another instance. Moderate dehydration can be readily removed by scraping with a blunt instrument.

Excessive dehydration masks the normal flesh color and penetrates the product. It affects more than 5% up to and including 10% of the surface area. If more than 10% of the surface area is affected, each additional 10% of surface area affected is another instance. Excessive dehydration requires a knife or other sharp instrument to remove.

#### 2. Uniformity of Block Size

This defect refers to the degree of conformity to the declared size. It includes deviations from the standard length, width, or thickness. Only one deviation for each dimension should be counted.

*Moderate:* A deviation of length and width of  $\frac{1}{8}$  inch (0.32 cm) or more up to and including  $\frac{1}{4}$  inch (0.64 cm). A deviation of thickness of  $\frac{1}{16}$  inch (0.16 cm) or more up to and including inch  $\frac{1}{8}$  (0.32 cm).

Excessive: If over  $\frac{1}{4}$  inch (0.64 cm), each additional inch (0.32 cm) of length and width is another instance. If over 1 inch (0.32 cm), each additional  $\frac{1}{16}$  inch (0.16 cm) of thickness is another instance.

## 3. Underweight

This defect refers to underweight deviations from the stated weight.

Slight. From 0.1 ounce (2.84 g) up to and including 1.0 ounce (28.35 g).

Moderate. Over 1.0 ounce (28.35 g) up to and including 4.0 ounces (113.4 g).

Excessive. If over 4.0 ounces (113.4 g), each additional 1.0 ounce (28.35 g) is another instance.

## 4. Angles

An acceptable edge angle is an angle formed by two adjoining surfaces whose apex (deviation from 90 degrees) is within 0.95 cm off a carpenter's square placed along its surfaces. An acceptable corner angle is an angle formed by three adjoining surfaces whose apex is within 0.95 cm of a carpenter's square.

#### 5. Improper Fill

This defect refers to voids, air packets, ice pockets, ragged edges, bumps, depressions, damage, and embedded packaging material, each of which is greater than  $\frac{1}{8}$  inch (0.32 cm) in depth and which would result in product loss after cutting. It is estimated by determining the minimum number of 1 ounce (28.35 g) model units that could be affected adversely. For the purpose of estimating product loss, the 1 ounce (28.35 g) model unit should have the dimensions  $4 \times 1 \times \frac{5}{8}$  inch (10.16  $\times$  2.54  $\times$  1.59 cm). The total number of model units that would be affected adversely is the number of instances.

### 6. Belly Flaps (Napes)

These may be either loose or attached to a fillet or part of a fillet. The maximum amount of belly flaps should not exceed 15% by declared weight of the block. If this amount does exceed 15%, each additional 5% by declared weight is another instance.

#### 7. Blood Spots

Each lump or mass of clotted blood greater than  $^3\!/_{16}$  inch (0.48 cm) up to and including  $^3\!/_{8}$  inch (0.95 cm) in any dimension is an instance. If a blood spot is larger than  $^3\!/_{8}$  inch (0.95 cm), each additional  $^3\!/_{16}$  (0.48 cm) is another instance.

#### 8. Bruises

Bruises include distinct, unnatural, dark, reddish, grayish, or brownish off-colors due to diffused blood. An instance is each bruise larger than 0.5 square inch (3.32 cm<sup>2</sup>) and less than 1.5 square inch (9.68 cm<sup>2</sup>). For each bruise 1.5 square inch (9.68 cm<sup>2</sup>) or larger, each additional complete 1.0 square inch (6.45 cm<sup>2</sup>) is another instance.

#### 9. Discoloration

Discoloration refers to deviations from reasonably uniform color characteristics of the species used, such as melanin deposits, yellowing, rusting, or other kinds of discoloration of the fish flesh.

*Moderate*. A noticeable but moderate degree which is greater than 0.5 square inch  $(3.23\,\mathrm{cm}^2)$  up to and including 1.5 square inch  $(9.68\,\mathrm{cm}^2)$  is one instance. If the discoloration is greater than 1.5 square inch  $(9.68\,\mathrm{cm}^2)$ , each additional complete 1.0 square inch  $(6.45\,\mathrm{cm}^2)$  is another instance.

*Excessive*. An excessive degree of discoloration which is greater than 0.5 square inch  $(3.23 \,\mathrm{cm}^2)$  up to and including 1.5 square inch  $(9.68 \,\mathrm{cm}^2)$  is one instance. If the discoloration is greater than 1.5 square inch  $(9.68 \,\mathrm{cm}^2)$  each additional complete 1.0 square inch  $(6.45 \,\mathrm{cm}^2)$  is another instance.

#### 10. Viscera, Roe, and Lace

Viscera and roe refer to any portion of the internal organs. Each occurrence of viscera and roe is an instance. Lace (frill) is a piece of tissue adhering to the edge of a flatfish (order Pleuronectifonnes) fillet. For each lace, each  $\frac{1}{2}$  inch (1.27 cm) is each instance.

#### 11. Skin

In skinless fish blocks, each piece of skin larger than 0.5 square inch (3.23 cm<sup>2</sup>) up to and including 1.0 square inch (6.45 cm<sup>2</sup>) is an instance. For each piece of skin that is larger than 1.0 square inch (6.45 cm<sup>2</sup>), each additional complete 0.5 square inch (3.23 cm<sup>2</sup>) in area is another instance. For pieces of skin smaller than 0.5 square inch (3.23 cm<sup>2</sup>), the number of 0.5 square inch (3.23 cm<sup>2</sup>) squares fully or partially occupied after collecting these pieces on a grid is the number of instances.

## 12. Membrane (black belly lining)

Each piece of membrane (black belly lining) larger than 0.5 square inch (3.23 cm<sup>2</sup>) up to and including 1.5 square inch (9.68 cm<sup>2</sup>) is an instance. For pieces of membrane (black belly lining) that are larger than 1.5 square inch (9.68 cm<sup>2</sup>), each additional complete 0.5 square inch (3.23 cm<sup>2</sup>) in area is another instance.

#### 13. Scales

For skin-on fillets that have been scaled, an instance is an area of scales over 0.5 square inch (3.23 cm<sup>2</sup>) up to and including 1.5 square inch (9.68 cm<sup>2</sup>). If the area is greater than 1.5 square inch (9.68 cm<sup>2</sup>), each additional complete 1.0 square inch (8.45 cm<sup>2</sup>) is another instance. Loose scales are counted and instances are deducted in the same manner as for skinless fillets. For skinless fillets, the first five to ten loose scales is an instance. If there are more than ten loose scales, each additional complete count of five loose scales is another instance.

#### 14. Foreign Material

Any harmless material not derived from fish, such as packaging material. Each occurrence is an instance.

#### 15. Bones (including pin bone and fin bone)

- 1. Each bone defect to a bone or part of a bone whose maximum profile is  $\frac{3}{16}$  inch (0.48 cm) or more in length, or at least  $\frac{1}{32}$  inch (0.08 cm) in shaft diameter or width, or, for bone chips, a longest dimension of at least  $\frac{3}{16}$  inch (0.48 cm).
- 2. An excessive degree of bone defect is each bone whose maximum profile cannot be fitted into a rectangle, drawn on a flat, solid surface, that has a length of  $1\frac{3}{16}$  inch (3.02 cm) and a width of  $\frac{3}{8}$  inch (0.95 cm).

#### 16. Fins or Part Fins

This defect refers to two or more bones connected by membrane, including internal or external bones, or both, in a cluster.

Moderate. Connected by membrane in a cluster, no internal bone.

Excessive. Connected by membrane in a cluster with internal bone.

#### 17. Parasites

Metazoan parasites. Each such parasite or fragment of such a parasite that is detected is an instance.

Parasitic copepods. Each such parasite or a fragment of such a parasite that is detected is an instance.

#### 18. Texture

Texture means that the cooked product has the textural characteristics of the indicated species of fish. It does not include any abnormal textural characteristics such as mushy, soft, gelatinous, tough, dry, or rubbery.

Moderate. Moderately abnormal textural characteristics.

Excessive. Excessively abnormal textural characteristics.

#### V. FROZEN MINCED FISH BLOCKS

## A. Product Description

Frozen minced fish blocks are uniformly shaped masses of cohering minced fish flesh. A block may contain flesh from a single species or a mixture of species with or without food additives. The minced flesh consists entirely of mechanically separated fish flesh processed and maintained in accordance with good commercial practice. This minced flesh is made entirely from species that are known to be safe and suitable for human consumption.

## B. Product Forms: Types

- Unmodified—no food additives used.
  - a. Single species
  - b. Mixed species
- 2. *Modified—contains food additives*.
  - a. Single species
  - b. Mixed species

### C. Color Classifications

- 1. White
- 2. Light
- 3. Dark

#### D. Texture

- 1. Coarse—Flesh has a fibrous consistency.
- 2. Fine—Flesh has a partially fibrous consistency because it is a mixture of small fibers and paste.
- 3. Paste/puree—Flesh has no fibrous consistency.

#### E. Definitions of Defects

Deteriorative color refers to discoloration from the normal characteristics of the material used. Deterioration can be due to yellowing of fatty material, to browning of blood pigments, or other changes.

- 1. Slight deteriorative discoloration—refers to a color defect that is slightly noticeable but does not seriously affect the appearance, desirability, or eating quality of the product.
- 2. Moderate deteriorative discoloration—refers to a color defect that is conspicuously noticeable but does not seriously affect the appearance, desirability, or eating quality of the product.
- 3. Excessive deteriorative discoloration—refers to a defect that is conspicuously noticeable and that seriously affects the appearance, desirability, or eating quality of the product.

*Dehydration* refers to a loss of moisture from the surfaces of the product during frozen storage.

- 1. Slight dehydration is surface color masking, affecting more than 5% of the area, which can be readily removed by scraping with a blunt instrument.
- 2. Moderate dehydration is deep color masking penetrating the flesh, affecting less than 5% of the area, and requiring a knife or other sharp instrument to remove.
- 3. Excessive dehydration is deep color masking penetrating the flesh, affecting more than 5% of the area, and requiring a knife or other sharp instruments to remove.

Uniformity of size refers to the degree of conformity to the declared contracted dimensions of the blocks. A deviation is considered to be any deviation from the contracted length, width, or thickness; or from the average dimensions of the blocks, physically determined, if no dimensions are contracted. Only one deviation from each dimension may be assessed. Two readings for length, three readings for width, and four readings for thickness will be measured.

- 1. Slight—two or more deviations from declared or average length, width, and thickness up to  $\pm \frac{1}{8}$  inch.
- 2. Moderate—two or more deviations from declared or average length, width, and thickness from  $\pm \frac{1}{8}$  inch to  $\pm xx$  inch (variable, depending on product).
- 3. Excessive—two or more deviations from declared or average length, width, and thickness over  $\pm \frac{3}{8}$  inch.

*Uniformity of weight* refers to the degree of conformity to the declared weight. Only underweight deviations are assessed.

- 1. Slight—any minus deviation of not more than 2 ounces.
- 2. Excessive—any minus deviation over 2 ounces.

Angles. An acceptable edge angle is an angle formed by two adjoining surfaces of the fish block whose apex is within  $\frac{3}{8}$  inch of a carpenter's square placed along the surfaces of the block. For each edge angle, three readings will be made and at least two readings must be acceptable for the whole edge angle to be acceptable. An acceptable corner angle is an angle formed by three adjoining surfaces whose apex is within  $\frac{3}{8}$  inch of the apex of a carpenter's square placed on the edge surfaces. Any edge or corner angle which fails to meet these measurements is unacceptable.

- 1. Slight—two unacceptable angles.
- 2. Moderate—three unacceptable angles.
- 3. Excessive—four or more unacceptable angles.

Improper fill refers to surface and internal air or ice voids, ragged edges, or damage. Improper fill is measured as the minimum number of 1 ounce units that would be adversely affected when the block is cut. For this purpose, the dimensions of a 1 ounce unit are  $4 \times 1 \times \frac{5}{8}$  inch.

- 1. Slight—one to three units adversely affected.
- 2. Excessive—over three units adversely affected.

*Blemishes* refer to pieces of skin, scales, blood spots, nape (belly) membranes (regardless of color), or other harmless extraneous material. One instance means that the area occupied by a blemish or blemishes is equal to a  $\frac{1}{4}$  inch square. Instances are prorated on a per pound basis.

- 1. Slight—5 to 15 instances per pound
- 2. Moderate—more than 15 but less than 30 instances per pound
- 3. Excessive—30 or more instances per pound

Bones refers to any objectionable bone or piece of bone that is  $\frac{1}{4}$  inch or longer and is sharp and rigid. Perceptible bones should also be checked by their grittiness during the normal evaluation of the texture of the cooked product (10). Bones are prorated on a five pound sample unit basis.

- 1. Slight—1 to 2 bones per five pound sample unit.
- 2. Moderate—3 to 4 bones per five pound sample unit.
- 3. Excessive—over 4 bones, but not to exceed 10 bones, per five pound sample unit.

Flavor and odor are evaluated organoleptically by smelling and tasting the product after it has been cooked.

Good flavor and odor (essential requirements for a Grade A product) means that the cooked product has the flavor and odor characteristic of the indicated species of fish and is free from staleness, bitterness, rancidity, and off-flavors and off-odors of any kind.

Reasonably good flavor and odor (minimum requirements of Grade B product) means that the cooked product is moderately absent of flavor and odor characteristic of the indicated species. The product is free from rancidity, bitterness, staleness, and off-flavors and off-odors of any kind.

Minimal acceptable flavor and odor (minimum requirements of a Grade C product) means that the cooked product has moderate storage induced flavor and odor but is free from any objectionable off-flavors and off-odors that may be indicative of spoilage or decomposition.

Texture defects are judged on a sample of the cooked fish.

- 1. Slight—flesh is fairly firm, only slightly spongy or rubbery. It is not mushy. There is no grittiness due to bone fragments.
- 2. Moderate—flesh is mildly spongy or rubbery. Slight grittiness may be present due to bone fragments.
- 3. Excessive—flesh is definitely spongy, rubbery, very dry, or very mushy. Moderate grittiness may be present due to bone fragments.

#### F. Additives

Minced fish blocks may be modified with food additives as necessary to stabilize product quality in accordance with the federal requirements.

## G. Hygiene

The fish material should be processed and maintained in accordance with federal requirements.

#### VI. FROZEN RAW FISH PORTIONS

## A. Description of the Product

The product described in this part consists of clean, wholesome, shaped masses of cohering pieces (not ground) of fish flesh. The fish portions are cut from frozen fish blocks, and are packaged in accordance with good manufacturing practice. They are maintained at temperatures necessary for the preservation of the product. All fish portions in an individual package are prepared from the flesh of one species of fish.

## B. Styles of Frozen Raw Fish Portions

#### 1. Style I—Skinless Portions

Portions prepared from fish blocks which have been made with skinless fillets.

#### 2. Style II—Skin-on Portions

Portions prepared from fish blocks that have been made from demonstrably acceptable skin-on fillets.

#### C. Types

#### 1. Type I—Uniform Shaped

All portions in the sample are uniformly shaped.

#### 2. Type II—Specialty Cut

All portions not covered in Type I.

#### D. Definitions of Defects

Dehydration refers to the presence of dehydrated (water-removed) tissue in the portions. Slight dehydration is surface dehydration that is not color masking. Deep dehydration is color masking and cannot be removed by scraping with a blunt instrument.

*Uniformity of size* refers to the degree of uniformity in length and width of the frozen portions. Deviations are measured from the combined lengths of the two shortest and/or the combined widths of the two widest minus the combined widths of the two narrowest in the sample.

Uniformity of weight refers to the degree of uniformity of the weights of portions. Uniformity is measured by the combined weight of the two heaviest portions divided by the combined weight of the two lightest portions in the sample. No deductions are made for weight ratios less than 1.2 for Type I.

Blemishes refers to skin (except for Style II), blood spots or bruises, objectionable dark fatty flesh, or extraneous material. Instances of blemishes refer to each occurrence measured by placing a plastic grid marked off in  $\frac{1}{4}$  inch squares  $\frac{1}{16}$  square inch) over the defect area. Each square is counted as 1 whether it is full or fractional.

*Bones* means the presence of potentially harmful bones in a portion. A potentially harmful bone is one that after being cooked is capable of piercing or hurting the palate.

Texture defects of the fish flesh and texture of skin in Style II refers to the absence of the normal textural properties of the cooked fish flesh and to the absence of tenderness of the cooked skin in Style II.

Normal textural properties of cooked fish flesh are tenderness, firmness, and moistness without excess water. Texture defects of the cooked flesh are dryness, mushiness, toughness, and rubberiness. Texture defects of the cooked skin in Style II are mushiness, rubberiness, toughness, and stringiness.

#### E. General Definitions

Small (overall assessment) refers to a condition that is noticeable but is only slightly objectionable.

Large (overall assessment) refers to a condition that not only is noticeable but is seriously objectionable.

Minor (individual assessment) refers to a defect that slightly affects the appearance and/or utility of the product.

Major (individual assessment) refers to a defect that seriously affects the appearance and/or utility of the product.

Net weight: The net weight of the portions if glazed should be determined by the following method:

- 1. Weigh the portions with the glaze intact, which gives the gross weight.
- 2. Thaw the glaze from the surfaces of the product with flowing tap water.
- 3. Gently wipe off the excess water from the surfaces with a single water-saturated paper towel.
- 4. Weigh the deglazed portions, which gives the net weight.

#### VII. FROZEN RAW BREADED FISH STICKS

#### A. Description of the Product

Frozen raw breaded sticks are clean, wholesome, rectangular-shaped unglazed masses of cohering pieces (not ground) of fish flesh coated with breading. The sticks are cut from frozen fish blocks; are coated with a suitable, wholesome batter and breading; are packaged, and frozen in accordance with good commercial practice. They are maintained at temperatures necessary for preservation of the product. Frozen raw breaded fish sticks weigh up to and including  $1\frac{1}{2}$  ounces; are at least  $\frac{3}{8}$  inch thick; and their largest dimension is at least 3 times the next largest dimension. All sticks in an individual package are prepared from the flesh of one species of fish.

### B. Composition of the Product

Frozen raw breaded fish sticks should contain 72% by weight of fish flesh determined by the official end-product method. Fish flesh content may be determined by the on-line

method, provided that the results are consistent with the fish flesh content requirement of 72% by weight when verified by the official end-product method. Production methods employed in official establishments should be kept relatively constant for each production lot so as to minimize variation in any factors that may affect the relative fish flesh content.

#### C. Definitions

Selection of the sample unit: The sample unit should consist of 10 frozen raw breaded fish sticks taken at random from one or more packages as required. The fish sticks are spread out on a flat pan or sheet and are examined.

#### 1. Examination of Sample—Frozen State

Condition of package refers to the presence in the package of loose breading and/or loose frost.

Ease of separation refers to the difficulty of separating sticks from each other or from packaging material that are frozen together during the freezing.

Broken stick means a stick with a break or cut equal to or greater than one-half the width of the stick.

Damaged stick means a stick that has been mashed, physically or mechanically injured, misshaped, or mutilated to the extent that its appearance is materially affected. The amount of damage is measured by using a grid composed of squares of  $\frac{1}{4}$  inch (that is, squares with an area of  $\frac{1}{16}$  square inch each) to measure the area of the stick affected. Deductions are not made for damage less than  $\frac{1}{16}$  square inch.

Uniformity of size refers to the degree of uniformity in length and width of the frozen sticks. Deviations are measured from the combined lengths of the two longest minus the combined lengths of the two shortest and/or the combined widths of the two widest minus the combined widths of the two narrowest. Deductions are not made for overall deviations in length or width up to  $\frac{1}{4}$  inch.

Uniformity of weight refers to the degree of uniformity of the weights of the sticks. Uniformity is measured by the combined weight of the two heaviest sticks divided by the combined weight of the two lightest sticks.

No deductions are made for weight ratios less than 1.15.

Cooked state means the state of the product after cooking in accordance with the instructions accompanying the product. However, if specific instructions are lacking, the product for inspection is cooked as follows: Transfer the product, while still in the frozen state, into a wire mesh fry basket large enough to hold the fish sticks in a single layer and cook by immersing them for 2–3 minutes in 375°F liquid or hydrogenated cooking oil. After cooking, allow the fish sticks to drain 15 seconds and place the fish sticks on a paper napkin or towel to absorb excess oil.

#### 2. Examination of Sample—Cooked State

Distortion refers to the degree of bending of the long axis of the stick. Distortion is measured as the greatest deviation from the long axis. Deductions are not made for deviations of less than  $\frac{1}{4}$  inch.

Coating defects refers to breaks, lumps, ridges, depressions, blisters, or swells and curds in the coating of the cooked product. Breaks in the coating are objectionable bare spots through which the fish flesh is plainly visible. Lumps are objectionable outcroppings of breading on the stick surface.

Ridges are projections of excess breading at the edges of the fish flesh.

Depressions are objectionable visible voids or shadow areas that are lightly covered by breading. Blisters are measured by the swelling or exposed area in the coating resulting from the bursting or breaking of the coating. Curd refers to craterlike holes in the breading filled with coagulated albumin. Instances of these defects are measured by a plastic grid marked off in  $\frac{1}{4}$ -inch squares ( $\frac{1}{16}$  square inch). Each square is counted as 1 whether it is full or fractional.

Blemishes refers to skin, blood spots or bruises, objectionable dark fatty flesh, or extraneous material. Instances of blemishes refers to each occurrence measured by placing a plastic grid marked off in  $\frac{1}{4}$ -inch squares ( $\frac{1}{16}$  square inch) over the defect area. Each square is counted as 1 whether it is full or fractional.

*Bones* means the presence of potentially harmful bones in a stick. A potentially harmful bone is one that after being cooked is capable of piercing or hurting the palate.

Texture defects of the coating refers to the absence of the normal textural properties of the coating, which are crispness and tenderness. Coating texture defects are dryness, sogginess, mushiness, doughyness, toughness, pastiness as sensed by starchiness or other sticky properties felt by mouth tissues, and/or mealiness.

Texture defects of the fish flesh refers to the absence of the normal textural properties of the cooked fish flesh which are tenderness, firmness, and moistness without excess water. Texture defects of the flesh are dryness, mushiness, toughness, and rubberyness.

#### VIII. FROZEN RAW BREADED FISH PORTIONS

## A. Description of the Product

Frozen raw breaded portions are clean, wholesome, uniformly shaped, unglazed masses of cohering pieces (not ground) of fish flesh coated with breading. The portions are cut from frozen fish blocks; are coated with a suitable, wholesome batter and breading; and are packaged and frozen in accordance with good commercial practice. They are maintained at temperatures necessary for the preservation of the product.

Frozen raw breaded fish portions weigh more than  $1\frac{1}{2}$  ounces, and are at least  $\frac{3}{8}$ -inch thick. Frozen raw breaded fish portions contain not less than 75%, by weight, of fish flesh. All portions in an individual package are prepared from the flesh of one species of fish.

#### B. Styles of Frozen Raw Breaded Fish Portions

#### 1. Style I—Skinless Portions

Portions prepared from fish blocks that have been made with skinless fillets.

## 2. Style II—Skin-on Portions

Portions prepared from fish blocks that have been made with demonstrably acceptable skin-on fillets.

#### C. Composition of the Product

1. Frozen raw breaded fish portions should contain 75% by weight of fish flesh Fish flesh content may be determined by the on-line method, provided that the results are consistent

with the fish flesh content requirement of 75% by weight, when verified by the official end-product method.

2. Production methods employed in official establishments should be kept relatively constant for each production lot so as to minimize variation in any factors that may affect the relative fish flesh content.

## 1. Examination of Sample—Frozen State

Condition of package refers to the presence in the package of loose breading and/or loose frost.

Ease of separation refers to the difficulty of separating the portions from each other or from the packaging material.

*Broken portion* means a portion with a break or cut equal to or greater than one-half the width or length of the portion.

Damaged portion means a portion that has been mashed, physically or mechanically injured, misshaped, or mutilated to the extent that its appearance is materially affected. The amount of damage is measured by using a grid composed of squares  $\frac{1}{4} \times \frac{1}{4}$ -inch (that is, squares with an area of  $\frac{1}{16}$  square inch each) to measure the area of the portion affected. No deductions are made for damage of less than  $\frac{1}{16}$  square inch.

Uniformity of size refers to the degree of uniformity in length and width of the frozen portions. Deviations are measured from the combined lengths of the two longest minus the combined lengths of the two shortest and/or the combined widths of the two widest minus the combined widths of the two narrowest portions in the sample. Deductions are not made for overall deviations in length or width up to  $\frac{1}{4}$  inch.

Uniformity of weight refers to the degree of uniformity of the weights of the portions. Uniformity is measured by the combined weight of the two heaviest portions divided by the combined weight of the two lightest portions in the sample. No deductions are made for weight ratios less than 1.2.

Cooked state means the state of the product after being cooked in accordance with the instructions accompanying the product.

#### 2. Examination of Sample—Cooked State

Distortion refers to the degree of bending of the long axis of the portion. Distortion is measured as the greatest deviation from the long axis. Deductions are not made for deviations of less than  $\frac{1}{4}$  inch.

Coating defects refers to breaks, lumps, ridges, depressions, blisters or swells, and curds in the coating of the cooked product. Breaks in the coating are objectionable bare spots through which the fish flesh is plainly visible. Lumps are objectionable outcroppings of breading on the portion surface. Ridges are projections of excess breading at the edges of the portions.

Depressions are objectionable visible voids or shadow areas that are lightly covered by breading. Blisters are measured by the swelling or exposed area in the coating resulting from the bursting or breaking of the coating. Curd refers to craterlike holes in the breading filled with coagulated white or creamy albumin. Instances of these defects are measured by a plastic grid marked off in  $\frac{1}{4}$ -inch squares of ( $\frac{1}{16}$  square inch). Each square is counted as 1 whether it is full or fractional.

Blemishes refers to skin (except for Style II), blood spots or bruises, objectionable dark fatty flesh, or extraneous material. Instances of blemishes refers to each occurrence

measured by placing a plastic grid marked off in  $\frac{1}{4}$ -inch squares ( $\frac{1}{16}$  square inch) over the defect area. Each square is counted as 1 whether it is full or fractional.

*Bones* means the presence of potentially harmful bones in a portion. A potentially harmful bone is one that after being cooked is capable of piercing or hurting the palate.

Texture defects of the coating refers to the absence of the normal textural properties of the coating, which are crispness and tenderness. Defects in coating texture are dryness, sogginess, mushiness, doughyness, toughness, pastyness, as sensed by starchiness or other sticky properties felt by mouth tissues, and/or mealiness.

Texture defects of the fish flesh and texture of skin in Style II refers to the absence of the normal textural properties of the cooked fish flesh and to the absence of tenderness of the cooked skin in Style II.

Normal textural properties of cooked fish flesh are tenderness, firmness, and moistness without excess water. Texture defects of the cooked flesh are dryness, mushiness, toughness, and rubberyness. Texture defects of the cooked skin in Style II are mushiness. rubberyness, toughness, and stringiness.

Minimum fish flesh content—End-product determination refers to the minimum percent, by weight, of the average fish flesh content of three frozen raw breaded portions (sample unit for fish flesh determination).

## IX. FROZEN FRIED FISH STICKS

## A. Description of the Product

Frozen fried fish sticks are clean wholesome, rectangular unglazed masses of cohering pieces (not ground) of fish flesh coated with breading and partially cooked. The sticks are cut from frozen fish blocks; are coated with a suitable wholesome batter and breading; are fried, packaged, and frozen in accordance with good manufacturing practices. They are maintained at temperatures necessary for preservation of the product. Frozen fried fish sticks weigh up to and including  $1\frac{1}{2}$  ounces; are at least three-eighths of an inch thick; and their largest dimension is at least three times the next largest dimension. All sticks in an individual package are prepared from the flesh of one species of fish.

## B. Composition of the Product

Frozen fried fish sticks should contain 60% by weight of fish flesh. Fish flesh content may be determined by the on-line method, provided that the results are consistent with the fish flesh content requirement of 60% by weight, when verified by the official end-product method.

Production methods employed in official establishments should be kept relatively constant for each production lot so as to minimize variation in any factors that may affect the relative fish flesh content.

Definitions of factors for point deductions are as follows:

#### 1. Examination of Sample—Frozen State

Condition of package refers to the presence in the package of free excess oil and/or loose breading and/or loose frost.

Ease of separation refers to the difficulty of separating sticks from each other or from packaging material that are frozen together after the frying operation and during the freezing.

Broken stick means a stick with a break or cut equal to or greater than one-half the width of the stick.

Damaged stick means a stick that has been mashed, physically or mechanically injured, misshaped or mutilated to the extent that its appearance is materially affected. The amount of damage is measured by using a grid composed of squares  $\frac{1}{4}$  inch (that is, squares with an area of  $\frac{1}{16}$  square inch each) to measure the area of the stick affected. Deductions are not made for damage less than  $\frac{1}{16}$  square inch.

Uniformity of size refers to the degree of uniformity in length and width of the frozen sticks. Deviations are measured from the combined lengths of the two longest minus the combined lengths of the two shortest and/or the combined widths of the two widest minus the combined widths of the two narrowest. Deductions are not made for overall deviations in length of width up to  $\frac{1}{4}$  inch.

Uniformity of weight refers to the degree of uniformity of the weights of the sticks. Uniformity is measured by the combined weight of the two heaviest sticks divided by the combined weight of the two lightest sticks. No deductions are made for weight ratios less than 1.15.

Cooked state means the state of the product after cooking in accordance with the instructions accompanying the product.

## 2. Examination of Sample—Cooked State

Distortion refers to the degree of bending of the long axis of the stick. Distortion is measured as the greatest deviation from the long axis. Deductions are not made for deviations of less than  $\frac{1}{4}$  inch.

Coating defects refers to breaks, lumps, ridges, depressions, blisters or swells and curds in the coating of the cooked product. Breaks in the coating are objectionable bare spots through which the fish flesh is plainly visible. Lumps are objectionable outcroppings of breading on the stick surface.

Ridges are projections of excess breading at the edges of the fish flesh.

Depressions are objectionable visible voids or shadow areas that are lightly covered by breading. Blisters are measured by the swelling or exposed area in the coating resulting from the bursting or breaking of the coating. Curd refers to craterlike holes in the breading filled with coagulated albumin. Instances of these defects are measured by a plastic grid marked off in  $\frac{1}{4}$  inch squares ( $\frac{1}{16}$  square inch). Each square is counted as one whether it is full or fractional.

*Blemishes* refers to skin, blood spots, or bruises, objectionable dark fatty flesh, carbon specks or extraneous material. Instances of blemishes refers to each occurrence measured by placing a plastic grid marked off in  $\frac{1}{4}$  inch squares ( $\frac{1}{16}$  square inch) over the defect area. Each square is counted as one whether it is full or fractional.

*Bones* means the presence of potentially harmful bones in a stick. A potentially harmful bone is one that after being cooked is capable of piercing or hurting the palate.

Texture defects of the coating refers to the absence of the normal textural properties of the coating, which are crispness and tenderness. Coating texture defects are dryness, sogginess, mushiness, doughyness, toughness, pastyness, as sensed by starchiness or other sticky properties felt by mouth tissues, oiliness to the degree of impairment of texture, and/ or mealiness.

Texture defects of the fish flesh refers to the absence of normal textural properties of the cooked fish flesh, which are tenderness, firmness, and arid moistness without excess water. Texture defects of the flesh are dryness, softness, toughness, and rubberyness.

#### X. FROZEN FRIED FISH PORTIONS

## A. Description of the Product

Frozen fried fish portions are clean, wholesome, uniformly shaped, unglazed masses of cohering pieces (not ground) of fish flesh coated with breading and partially cooked. The portions are cut from frozen fish blocks; coated with a suitable, wholesome batter and breading; are fried, packaged, and frozen in accordance with good manufacturing practices. They are maintained at temperatures necessary for preservation of the product. Frozen fried fish portions weigh more than  $1\frac{1}{2}$  ounces and are at least three-eighths of an inch thick. All portions in an individual package are prepared from the flesh of one species of fish.

## **B.** Composition of the Product

Frozen fried fish portions should contain 65% by weight of fish flesh. Fish flesh content may be determined by the on-line method, provided that the results are consistent with the fish flesh content requirement of 65% by weight, when verified by the official end-product method.

Production methods employed in official establishments should be kept relatively constant for each production lot so as to minimize variation in any factors that may affect the relative fish flesh content.

#### 1. Examination of Sample—Frozen State

Condition of package refers to the presence in the package of free excess oil and/or loose breading and/or loose frost.

Ease of separation refers to the difficulty of separating portions from each other or from packaging material that are frozen together after the frying operation and during the freezing.

*Broken portion* means a portion with a break or cut equal to or greater than one-half the width or length of the portion.

Damaged portion means a portion that has been mashed, physically or mechanically injured, misshaped or mutilated to the extent that its appearance is materially affected. The amount of damage is measured by using a grid composed of squares  $\frac{1}{4}$  inch (that is, squares with an area of  $\frac{1}{16}$  square inch each) to measure the area of the portion affected. Deductions are not made for damage less than  $\frac{1}{16}$  square inch.

Uniformity of size refers to the degree of uniformity in length and width of the frozen portions. Deviations are measured from the combined lengths of the two longest minus the combined lengths of the two shortest and/or the combined widths of the two widest minus the combined widths of the two narrowest. Deductions are not made for overall deviations in length or width up to  $\frac{1}{4}$  inch.

*Uniformity of weight* refers to the degree of uniformity of the weights of the portions. Uniformity is measured by the combined weight of the two heaviest portions divided by

the combined weight of the two lightest portions. No deductions are made for weight ratios less than 1.20.

Cooked state means the state of the product after cooking in accordance with the instructions accompanying the product.

#### XI. FRESH AND FROZEN SHRIMP

## A. Product Description

The products are clean wholesome shrimp that are fresh or frozen, raw or cooked. Product forms are

## B. Types

- 1. Chilled, fresh (not previously frozen)
- 2. Unfrozen, thawed (previously frozen)
- 3. Frozen individually (IQF), glazed or unglazed
- 4. Frozen solid pack, glazed or unglazed

## C. Styles of Fresh and Frozen Shrimp

Raw (uncoagulated protein)

## D. Blanched (parboiled)

Heated for a period of time such that the surface of the product reaches a temperature adequate to coagulate the protein.

Cooked—heated for a period of time such that the thermal center of the product reaches a temperature adequate to coagulate the protein.

## E. Market Forms

- 1. Heads on (head, shell, tail fins on)
- 2. Headless (only head removed: shell, tail fins on)
- 3. Peeled, undeveined, round, tail on (all shell removed except last shell segment and tail fins, with segments unslit)
- 4. Peeled, undeveined, round, tail off (all shell and tail fins removed, with segments unslit)
- 5. Peeled and deveined, round, tail on (all shell removed except last shell segment and tail fins, with segments shallowly slit to last segment)
- 6. Peeled and deveined, round, tail off (all shell and tail fins removed, with segments shallowly slit to last segment)
- 7. Peeled and deveined, fantail or butterfly, tail on (all shell removed except last shell segment and tail fins, with segments deeply slit to last segment)
- 8. Peeled and deveined, fantail or butterfly, tail off (all shell and tail fin removed, with segments deeply slit to last segment)
- 9. Peeled and deveined, western (all shell removed except last shell segment and tail fins, with segments split to fifth segment and vein removed to end of cut)
- 10. Other forms of shrimp as specified and so designated on the label

#### F. Examination in the Frozen State

Dehydration refers to a general drying of the shrimp flesh that is noticeable after any glaze and shell are removed. It includes any detectable change from the normal characteristic, bright appearance of freshly caught, properly iced, or properly processed shrimp.

*Slight dehydration* means scarcely noticeable drying of the shrimp flesh that will not affect the sensory quality of the sample.

Moderate dehydration means conspicuous drying of the shrimp flesh that will not seriously affect the sensory quality of the sample.

Excessive dehydration means conspicuous drying that will seriously affect the sensory quality of the sample.

#### G. Examination in the Fresh or Thawed State

Uniformity of size refers to the degree of uniformity of the shrimp in the container to determine their conformity to the declared count.

Black spots, improperly headed (throats), and improperly cleaned ends refer to the presence of any objectionable black or darkened area that affects the desirability or sensory quality of the shrimp, whether the market form is shell-on or peeled. Objectionable black spot refers to more than three instances of penetrating black spot that is visible but difficult to measure because of its small size (approximately the size of a pencil point), or any areas larger than a pencil point that penetrates the flesh, or aggregate areas of nonpenetrating surface black spot on the shell or membrane that is equal to or greater than  $\frac{1}{3}$  the area of the smallest segment.

Assessments are made on individual shrimp.

Throats are those portions of flesh and/or extraneous material from the head (cephalothorax) that remain attached to the first segment after heading.

## H. Pieces of Shrimp, Broken or Damaged Shrimp

*Piece* means for a count of 70 or less unglazed shrimp per pound (0.45 kg), any shrimp that has fewer than five segments, with or without tail fins attached; or, for a count of more than 70 unglazed shrimp per pound (0.45 kg), any shrimp that has fewer than four segments; or any whole shrimp with a break in the flesh greater than  $\frac{2}{3}$  of the thickness of the shrimp where the break occurs.

Broken shrimp means a shrimp having a break in the flesh greater than  $\frac{1}{3}$  of the thickness of the shrimp.

Damaged shrimp means a shrimp that is crushed or mutilated so as to materially affect its appearance or usability.

Unusable material includes the following:

Legs refer to walking legs only, whether attached or not attached to the body (heads-on market form excepted).

Loose shell and antennae are any pieces of shell or antennae that are completely detached from the shrimp.

Flipper refers to any detached tail fin with or without the last shell segment attached, with or without flesh inside.

Extraneous material means any harmless material in a sample unit that is not shrimp material.

## I. Unacceptable Shrimp and Heads

Unacceptable shrimp refers to abnormal or diseased shrimp.

Head refers to the cephalothorax, except for heads-on shrimp.

Inadvertently peeled and improperly peeled shrimp refer to the presence or absence of head, shell segment, swimmeret, or tail fin, which should or should not have been removed from certain market forms (shell-on shrimp with tail fins and/or telson missing is inadvertently peeled, but if the last segment of flesh is missing, the shrimp is damaged).

Improperly deveined shrimp refers to the presence of dark vein (alimentary canal) containing sand or sediment; or roe that should have been removed for peeled and deveined market forms. For shrimp of 70 count per pound (0.45 kg) or less, aggregate areas of dark vein or roe that are longer than one segment are defect. For shrimp of 71 to 500 count per pound (0.45 kg) or less, aggregate areas of dark vein or roe defect that are longer than two segments are a defect.

*Note*: This does not pertain to the last segment. For shrimp of over 500 count per pound (0.45 kg), dark vein or roe of any length is not a defect.

#### J. Examination in the Cooked State

The texture of cooked shrimp should be firm, slightly resilient but not tough, moist but not mushy. Texture as a defect refers to an undesirable toughness, dryness, or mushiness that deviated from the normal characteristics of the species when freshly caught, properly processed, and cooked.

Slight. Slightly tough, dry, but not mushy.

Moderate. Moderately tough, dry, or mushy.

Excessive. Excessively tough, very dry, or very mushy.

#### XII. FROZEN RAW BREADED SHRIMP

The FDA has provided the following on the standards for frozen raw breaded shrimp.

#### A. Description

Frozen raw breaded shrimp are whole, clean, wholesome, headless, peeled shrimp that have been deveined where applicable of the regular commercial species, and coated with a wholesome, suitable batter and/or breading. Whole shrimp consist of five or more segments of unmutilated shrimp flesh. They are prepared and frozen in accordance with good manufacturing practice and are maintained at temperatures necessary for the preservation of the product.

Frozen raw breaded shrimp is the food prepared by coating one of the optional forms of shrimp with safe and suitable batter and breading ingredients. The food is frozen.

The food tests not less than 50% of shrimp material as determined by a prescribed method

The term *shrimp* means the tail portion of properly prepared shrimp of commercial species. Except for composite units, each shrimp unit is individually coated. The optional forms of shrimp are

Fantail or butterfly. Prepared by splitting the shrimp. The shrimp are peeled, except that tail fins remain attached and the shell segment immediately adjacent to the tail fins may be left attached.

Butterfly, tail off. Prepared by splitting the shrimp; tail fins and all shell segments are removed.

Round. Round shrimp, not split; the shrimp are peeled, except that tail fins remain attached and the shell segment immediately adjacent to the tail fins may be left attached.

Round, tail off. Round shrimp, not split; tail fins and all shell segments are removed. Pieces. Each unit consists of a piece or a part of a shrimp; tail fins and all shell segments are removed.

The above information is categorized as follows.

## B. Styles of Frozen Raw Breaded Shrimp

## 1. Style I

Regular breaded shrimp are frozen raw breaded shrimp containing a minimum of 50% of shrimp material.

#### 2. Style II

Lightly breaded shrimp are frozen raw breaded shrimp containing a minimum of 65% of shrimp material.

#### C. Types

## 1. Type I—Breaded Fantail Shrimp

Subtype A. Split (butterfly) shrimp with the tail fin and the shell segment immediately adjacent to the tail fin.

Subtype B. Split (butterfly) shrimp with the tail fin but free of all shell segments.

Subtype C. Split (butterfly) shrimp without attached tail fin or shell segments.

#### 2. Type II—Breaded Round Shrimp

Subtype A. Round shrimp with the tail fin and the shell segment immediately adjacent to the tail fin.

Subtype B. Round shrimp with the tail fin but free of all shell segments.

Subtype C. Round shrimp without attached tail fin or shell segments.

## 3. Type III—Breaded Split Shrimp

## D. Definitions and Methods of Analysis

#### 1. Fantail Shrimp

This type is prepared by splitting and peeling the shrimp, except that for Subtype A, the tail fin remains attached and the shell segment immediately adjacent to the tail fin remains attached.

For subtype B, the tail fin remains, but the shrimp are free of all shell segments.

For subtype C, the shrimp are free of tail fins and all shell segments.

#### 2. Round Shrimp

This type is the round shrimp, not split. The shrimp are peeled except that for Subtype A, the tail fin remains attached and the shell segment immediately adjacent to the tail fin remains attached.

For subtype B, the tail fin remains, but the shrimp are free of all shell segments. For subtype C, the shrimp are free of all shell segments and tail fins.

## E. Composite Units

Each unit consists of two or more whole shrimp or pieces of shrimp, or both, formed and pressed into composite units prior to coating; tail fins and all shell segments are removed; large composite units, prior to coating, may be cut into smaller units.

The batter and breading ingredients referred to are the fluid constituents and the solid constituents of the coating around the shrimp. These ingredients consist of suitable substances that are not food additives as defined by regulations. If they are food additives as so defined, they are used in conformity with regulations established. Batter and breading ingredients that perform a useful function are regarded as suitable, except that artificial flavorings, artificial sweeteners, artificial colors, and chemical preservatives, other than those specifically permitted, are not suitable ingredients of frozen raw breaded shrimp. Chemical preservatives that are suitable are

- 1. Ascorbic acid, which may be used in a quantity sufficient to retard development of dark spots on the shrimp
- 2. The antioxidant preservatives listed in the regulations that may be used to retard development of rancidity of the fat content of the food, in amounts within the limits prescribed.

The label should name the food, as prepared from each of the optional forms of shrimp specified, and following the numbered sequence of the following:

- 1. "Breaded fantail shrimp." The word "butterfly" may be used in lieu of "fantail" in the name.
- 2. "Breaded butterfly shrimp, tail off."
- 3. "Breaded round shrimp."
- 4. "Breaded round shrimp, tail off."
- 5. "Breaded shrimp pieces."
- 6. Composite units.

If the composite units are in a shape similar to that of breaded fish sticks the name is "Breaded shrimp sticks"; if they are in the shape of meat cutlets, the name is "Breaded shrimp cutlets".

If prepared in a shape other than that of sticks or cutlets, the name is "Breaded shrimp—," the blank to be filled in with the word or phrase that accurately describes the shape, and which is not misleading.

The word "prawns" may be added in parentheses immediately after the word "shrimp" in the name of the food if the shrimp are of large size; for example, "Fantail breaded shrimp (prawns)." If the shrimp are from a single geographical area, the adjectival designation of that area may appear as part of the name; for example, "Breaded Alaskan shrimp sticks."

The names of the optional ingredients used should be listed on the principal display panel or panels of the label with such prominence and conspicuousness as to render them likely to be read and understood by the ordinary individual under customary conditions of purchase. If a spice that also imparts color is used, it should be designated as "spice and coloring," unless the spice is designated by its specific name. If ascorbic acid is used to retard development of dark spots on the shrimp, it should be designated as "Ascorbic acid added as a preservative" or "Ascorbic acid added to retard discoloration of shrimp."

If any other antioxidant preservative is used, such preservative should be designated by its common name followed by the statement "Added as a preservative."

Frozen raw lightly breaded shrimp complies with the provisions of frozen raw breaded shrimp except that it contains not less than 65% of shrimp material and that in the name prescribed the word "lightly" immediately precedes the words "breaded shrimp."

Factors evaluated on unbreaded or thawed debreaded product. Factors affecting qualities that are measured on the product in the unbreaded or thawed debreaded state are degree of deterioration, dehydration, sand veins, black spot, extra shell, extraneous material, and swimmerets.

*Dehydration* refers to the occurrence of whitish areas on the exposed ends of the shrimp (due to the drying of the affected area) and to a generally desiccated appearance of the meat after the breading is removed.

Deterioration refers to any detectable change from the normal good quality of freshly caught shrimp. It is evaluated by noting in the thawed product deviations from the normal odor and appearance of freshly caught shrimp.

Extraneous material consists of nonedible material such as sticks, seaweed, shrimp thorax, or other objects that may be accidentally present in the package.

*Slight* refers to a condition that is scarcely noticeable but does affect the appearance, desirability, and/or eating quality of breaded shrimp.

*Moderate* refers to a condition that is conspicuously noticeable but that does not seriously affect the appearance, desirability, and/or eating quality of the breaded shrimp.

*Marked* refers to a condition that is conspicuously noticeable and that does seriously affect the appearance, desirability, and/or eating quality of the breaded shrimp.

Excessive refers to a condition that is very noticeable and is seriously objectionable.

Halo means an easily recognized fringe of excess batter and breading extending beyond the shrimp flesh and adhering around the perimeter or flat edges of a split (butterfly) breaded shrimp.

Balling up means the adherence of lumps of the breading material to the surface of the breaded coating, causing the coating to appear rough, uneven, and lumpy.

Holidays means voids in the breaded coating as evidenced by bare or naked spots.

Damaged frozen raw breaded shrimp means frozen raw breaded shrimp that have been separated into two or more parts or that have been crushed or otherwise mutilated to the extent that their appearance is materially affected.

Black spot means any blackened area that is markedly apparent on the flesh of the shrimp.

Sand vein means any black or dark sand vein that has not been removed, except for that portion under the shell segment adjacent to the tail fin when present.

Extra shell means any shell segment(s) or portion thereof, contained in the breaded shrimp, except the first segment adjacent to the tail fin for Type I, Subtype A, and Type II, Subtype A.

#### XIII. FROZEN RAW SCALLOPS

## A. Description of the Product

Frozen raw scallops are clean, wholesome, adequately drained, whole or cut adductor muscles of the scallop of the regular commercial species. The portion of the scallop used should be only the adductor muscle eye that controls the shell movement. Scallops should be washed, drained, packed, and frozen in accordance with good manufacturing practices and are maintained at temperatures necessary for the preservation of the product. Only scallops of a single species should be used within a lot.

## B. Styles of Frozen Raw Scallops

## 1. Style I

Solid pack scallops are frozen together into a solid mass.

- 1. Substyle a. Glazed
- 2. Substyle b. Not glazed

## 2. Style II

Individually quick frozen pack (IQF) scallops are individually quick frozen. Individual scallops can be separated without thawing.

- 1. Substyle a. Glazed
- 2. Substyle b. Not glazed

## C. Types

- 1. Type 1. Adductor muscle.
- 2. Type 2. Adductor muscle with catch (gristle or sweet meat) portion removed.

#### D. Definitions of Defects

Dehydration refers to the loss of moisture from the scallop surface during frozen storage. A small degree of dehydration is color masking but can be easily scraped off. A large degree of dehydration is deep and color masking and requires a knife or other instrument to scrape off.

Extraneous materials are pieces or fragments of undesirable material that are naturally present in or on the scallops and that should be removed during processing.

An instance of minor extraneous material includes but is not limited to each occurrence of intestines, seaweed, etc., and each aggregate of sand and grit up to  $\frac{1}{2}$  inch square and located on the scallop surface. Deduction points should be assessed for additional instances of intestines, seaweed, etc., and aggregates of sand and grit up to  $\frac{1}{2}$  inch square.

An instance of major extraneous material includes but is not limited to each instance of shell or aggregate of embedded sand or other extraneous embedded material that affects the appearance or eating quality of the product.

*Texture* refers to the firmness, tenderness, and moistness of the cooked scallop meat, which is characteristic of the species.

*Net weight* means the total weight of the scallop meats within the package after removal of all packaging materials, ice glaze, or other protective materials.

## XIV. FROZEN RAW BREADED SCALLOPS AND FROZEN FRIED SCALLOPS

## A. Product Description

## 1. Frozen Raw Breaded Scallops

Frozen raw breaded scallops are

- 1. Prepared from wholesome, clean, adequately drained, whole or cut adductor muscles of the scallop of the regular commercial species, or scallop units cut from a block of frozen scallops that are coated with wholesome batter and breading.
- 2. Packaged and frozen according to good commercial practice and maintained at temperatures necessary for preservation.
- 3. Composed of a minimum of 50% by weight of scallop meat.

## 2. Frozen Fried Scallops

Frozen fried scallops are

- 1. Prepared from wholesome, clean, adequately drained, whole or cut adductor muscles of the scallop of the regular commercial species, or scallop units cut from a block of frozen scallops that are coated with wholesome batter and breading.
- 2. Precooked in oil or fat.
- 3. Packaged and frozen according to good commercial practice and maintained at temperatures necessary for preservation.
- 4. Composed of a minimum of 50% by weight of scallop meat.

## B. Styles of Frozen Raw Breaded Scallops and Frozen Fried Scallops

The styles of frozen raw breaded scallops and frozen fried scallops include

#### 1. Style I—Random Pack

Scallops in a package are reasonably uniform in weight and/or shape. The weight or shape of individual scallops is not specified.

#### 2. Style II—Uniform Pack

Scallops in a package consist of uniform shaped pieces that are of specified weight or range of weights.

#### C. Types

- 1. Type 1. Adductor muscle
- 2. Type 2. Adductor muscle with catch (gristle or sweet meat) portion removed

#### D. Definitions of Defects

Appearance refers to the condition of the package and ease of separation in the frozen state and continuity and color in the cooked state.

Condition of the package refers to freedom from packaging defects and the presence in the package of oil, and/or loose breading, and/or frost. Deduction points are based on the degree of the improper condition as small or large.

Ease of separation refers to the difficulty of separating scallops that are frozen together after the frying operation and during freezing.

Continuity refers to the completeness of the coating of the product in the cooked state. Lack of continuity is exemplified by breaks, ridges, and/or lumps of breading. Each  $^{1}/_{16}$  square inch area of any break, ridge, or lump of breading is considered an instance of lack of continuity. Individual breaks, ridges, or lumps of breading measuring less than  $^{1}/_{16}$  square inch are not considered objectionable. Deduction points are based on the percentage of the scallops within the package that contain small and/or large instances of lack of continuity.

## E. Workmanship Defects

Workmanship defects refer to the degree of freedom from doubled and misshaped scallops and extraneous material. The defects of doubled and misshaped scallops are determined by examining the frozen product, while the defects of extraneous materials are determined by examining the product in the cooked state. Deduction points are based on the percentage by count of the scallops affected within the package.

*Doubled scallops*. Two or more scallops that are joined together during the breading and/or frying operations.

Misshaped scallops. Elongated, flattened, mashed, or damaged scallop meats.

Extraneous material. Extraneous are pieces or fragments of undesirable material that are naturally present in or on the scallops and that should be removed during processing.

Examples of minor extraneous material include intestines, seaweed, and each aggregate of sand and grit within an area  $\frac{1}{2}$ -inch square.

Examples of major extraneous material include shell, aggregate of embedded sand, or other extraneous embedded material that affects the appearance or eating quality of the product.

*Texture of the coating:* 

Firm or crisp, but not tough, pasty, mushy, or oily Moderately tough, pasty, mushy, or oily Excessively tough, pasty, mushy, or oily

Texture of the scallop meat:

Firm, but tender and moist Moderately tough, dry, and/or fibrous or mushy Excessively tough, dry, and/or fibrous or mushy

*Character* refers to the texture of the scallop meat and of the coating and the presence of gristle in the cooked state.

*Gristle*. Gristle (type 2 only) is the tough elastic tissue usually attached to the scallop meat. Each instance of gristle is an occurrence.

Texture refers to the firmness, tenderness, and moistness of the cooked scallop meat and to the crispness and tenderness of the coating of the cooked product. The texture of the scallop meat may be classified as a degree of mushiness, toughness, and fibrousness. The texture of the coating may be classified as a degree of pastiness, toughness, dryness, mushiness, or oiliness.

## XV. FROZEN NORTH AMERICAN FRESHWATER CATFISH AND CATFISH PRODUCTS

## A. Scope and Product Description

The descriptions apply to products derived from farm-raised catfish, or from those taken from rivers and lakes in North American freshwater. They are of the following common commercial species and hybrids thereof:

- 1. Channel catfish (*Ictalurus punctatus*)
- 2. White catfish (*Ictalurus catus*)
- 3. Blue catfish (*Ictalurus furcatus*)
- 4. Flathead catfish (*Pylodictis olivaris*)

Fresh products will be packaged in accordance with good commercial practices and maintained at temperatures necessary for the preservation of the product. Frozen products will be frozen to  $0^{\circ}F$  ( $-18^{\circ}C$ ) at their center (thermal core) in accordance with good commercial practices and maintained at temperatures of  $0^{\circ}F$  ( $-18^{\circ}C$ ) or less.

The product may contain bones when the principal display panel clearly shows that the product contains bones.

#### **B.** Product Presentation

Catfish products may be presented and labeled as follows:

Types: Fresh or frozen. Styles: Skin on or skinless.

Market forms include but are not limited to the following:

- 1. Headed and gutted.
- 2. Headed and dressed are headed and gutted usually with fins removed. This form may be presented with or without the dorsal spine and with or without the collar bone.
- 3. Whole fillets are practically boneless pieces of fish cut parallel to the entire length of the backbone with the belly flaps and with or without the black membrane.
- 4. Trimmed fillets are whole fillets without belly flaps.
- 5. Fillet strips are strips of fillets weighing not less than  $\frac{3}{4}$  ounce.
- 6. Steaks are units of fish not less than  $1\frac{1}{2}$  ounces in weight that are sawn or cut approximately perpendicular (30 degrees to 90 degrees) to the axial length or backbone. They have two reasonably parallel surfaces. The number of tail sections that may be included in the package must not exceed the number of fish cut per package.
- 7. Nuggets are pieces of belly flaps with or without black membrane and weighing not less than  $\frac{3}{4}$  ounce.

Bone classifications: Practically boneless fillet or bone-in (fillet cut, with bones).

*Dehydration* applies to all frozen market forms. It refers to the loss of moisture from the surface, resulting in a whitish, dry, or porous condition:

Slight: surface dehydration that is not color masking (readily removed by scraping) and affecting 3 to 10% of the surface area.

Moderate: deep dehydration that is color masking, cannot be scraped off easily with a sharp instrument, and affects more than one percent but not more than 10% of the surface area.

Excessive: deep dehydration that is color masking and cannot be easily scraped off with a sharp instrument and affects more than 10% of the surface area.

Condition of the product applies to all market forms. It refers to freedom from packaging defects, cracks in the surface of a frozen product, and excess moisture (drip) or blood inside the package. Deduction points are based on the degree of this defect.

Slight refers to a condition that is scarcely noticeable but that does not affect the appearance, desirability, or eating quality of the product.

Moderate refers to a condition that is conspicuously noticeable but that does not seriously affect the appearance, desirability, or eating quality of the product.

Excessive refers to a condition that is conspicuously noticeable and that does seriously affect the appearance, desirability, or eating quality of the product.

Discoloration applies to all market forms. It refers to colors not normal to the species. This may be due to mishandling or the presence of blood, bile, or other substances.

Slight:  $\frac{1}{16}$  square inch up to and including 1 square inch in aggregate area.

Moderate: greater than 1 square inch up to and including 2 square inches in aggregate area.

Excessive: over 2 square inches in aggregate area. Also, each additional complete one square inch is again assessed points under this category.

*Uniformity* will be assigned in accordance with weight tolerances as follows:

Weight of portion: 0.75 to 4.16 ounces

Moderate: Over ½ ounce but not over ¼ ounce above or below declared weight of portion

Excessive: In excess of  $\frac{1}{4}$  ounce above or below declared weight of portion 4.17 to 11.20 ounces

Moderate: Over  $\frac{1}{8}$  ounce but not over  $\frac{1}{2}$  ounce above or below declared weight of portion

Excessive: In excess of  $\frac{1}{2}$  ounce above or below declared weight of portion 11.21 to 17.30 ounces

Moderate: Over  $\frac{1}{8}$  ounce but not over  $\frac{1}{8}$  ounce above or below declared weight of portion

Excessive: In excess of  $\frac{1}{8}$  ounce above or below declared weight of portion

Skinning cuts applies to skinless market forms. It refers to improper cuts made during the skinning operation as evidenced by torn or ragged surfaces or edges, or gouges in the flesh which detract from a good appearance of the product.

Slight:  $\frac{1}{16}$  square inch up to and including 1 square inch in aggregate area.

Moderate: Over 1 square inch up to and including 2 square inches in aggregate area.

Excessive: Over 2 square inches in aggregate area. Also, each additional complete 1 square inch is again assessed points under this category.

Heading applies to the presence of ragged cuts or pieces of gills, gill cover, pectoral fins, or collar bone after heading. Deduction points also will be assigned when the product is presented with the collar bone and it has been completely or partially removed.

Slight:  $\frac{1}{16}$  square inch up to and including 1 square inch in aggregate area.

Moderate: Over 1 square inch up to and including 2 square inches in aggregate area. Excessive: Over 2 square inches in aggregate area. Also, each additional complete one square inch is again assessed points under this category.

Evisceration applies to all market forms. It refers to the proper removal of viscera, kidney, spawn, blood, reproductive organs, and abnormal fat (leaf). The evisceration cut should be smooth and clean. Deduction points are based on the degree of defect.

Slight:  $\frac{1}{16}$  square inch up to and including 1 square inch in aggregate area.

Moderate: Over 1 square inch up to and including 2 square inches in aggregate area.

Excessive: Over 2 square inches in aggregate area. Also, each additional complete one square inch is again assessed points under this category.

Fins refer to the presence of fins, pieces of fins, or dorsal spines. It applies to all market forms except headed and gutted or headed and dressed catfish or catfish steaks. Deduction points also will be assigned when the product is intended to have the dorsal spine but it has been completely or partially removed.

Slight: Aggregate area up to and including 1 square inch.

Moderate: Over 1 square inch area up to and including 2 square inches.

Excessive: Over 2 square inches in aggregate area. Also, each additional complete 1 square inch is again assessed points under this category.

Bones (including pin bone) apply to all fillet and nugget market forms. Each bone defect is a bone or part of a bone that is  $\frac{3}{16}$  inch or more at its maximum length or  $\frac{1}{32}$  inch or more at its maximum shaft width, or for bone chips, a length of at least  $\frac{1}{16}$  inch. An excessive bone defect is any bone that cannot be fitted into a rectangle with a length of  $\frac{19}{16}$  inch and a width of  $\frac{1}{8}$  inch. In market forms intended to contain bones, the presence of bones will not be considered a physical defect.

Skin refers to the presence of skin on skinless market forms. For semiskinned forms, a skin defect is the presence of the darkly pigmented outside layers. Points will be assessed for each aggregate area greater than  $\frac{1}{2}$  square inch up to and including 1 square inch.

Bloodspots refers to the presence of coagulated blood.

Bruises refers to softening and discoloration of the flesh. Both bloodspots and bruises apply to all market forms. Points will be assessed for each aggregate area of bloodspots or bruises greater than  $\frac{1}{2}$  square inch up to and including 1 square inch.

Foreign material refers to extraneous material, including packaging material, not derived from the fish that is found on or in the sample. Each occurrence will be assessed.

Texture applies to all market forms and refers to the presence of normal texture properties of the cooked fish flesh, i.e., tender, firm, and moist, without excess water. Texture defects are described as dry, tough, mushy, rubbery, watery, and stringy.

Moderate: Noticeably dry, tough, mushy, rubbery, watery, stringy.

Excessive: Markedly dry, tough, mushy, rubbery, watery, stringy.

# **22**

## **Frozen Vegetables: Product Descriptions**

## **Peggy Stanfield**

Dietetic Resources, Twin Falls, Idaho, U.S.A.

#### I. INTRODUCTION

This book is not the proper forum for discussing the manufacture of every processed vegetable available in the market. However, regulatory agencies such the U.S. Department of Agriculture (USDA) and the U.S. Food and Drug Administration (FDA) have issued some minimal criteria for each processed vegetable such as what they are, what types and styles are available, and so on. The information in this chapter describes each available frozen vegetable product and has been modified from the product grades (USDA) and product standards (FDA). Product standards and product grades are established to achieve two objectives: to assure product safety and to minimize economic fraud.

The information provided here has one major objective: to remind a commercial processor of what each frozen vegetable is and of other applicable criteria for a particular product.

#### II. FROZEN ASPARAGUS

Frozen asparagus consists of sound and succulent fresh shoots of the asparagus plant (*Asparagus officinalis*). The product is prepared by sorting, trimming, washing, and blanching as necessary to assure a clean and wholesome product. It is then frozen and stored at temperatures necessary for preservation.

## A. Types

- 1. Green or all-green consists of units of frozen asparagus that are typically green, light-green, or purplish-green in color.
- 2. Green-white consists of frozen asparagus spears and tips that have typical green, light-green, or purplish-green color to some extent but which are white in the lower portions of the stalk.

## B. Styles

Spears or stalks style consists of units composed of the head and adjoining portion of the shoot that are 3 inches or more in length. Tips style consists of units composed of the head

and adjoining portion of the shoot that are less than 3 inches in length. Center cuts or cuts style consists of portions of shoots (with or without head material) that are cut transversely into units not less than one-half inch in length and that fail to meet the definition for cut spears or cuts and tips style.

Cut spears or cuts and tips style consists of the head and portions of the shoot cut transversely into units 2 inches or less but not less than one-half inch in length. To be considered this style, head material should be present in these amounts for the respective lengths of cuts:

- 1.  $1\frac{1}{4}$  inches or less. Not less than 18% (average), by count, of all cuts are head material.
- 2. Longer than  $1\frac{1}{4}$  inches. Not less than 25% (average), by count, of all cuts are head material.

#### III. FROZEN LIMA BEANS

Frozen lima beans are the frozen product prepared from the clean, sound, succulent seed of the lima bean plant without soaking, by shelling, washing, blanching, and properly draining. They are then frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation of the product.

## A. Types

- 1. Thin-seeded such as Henderson, Bush, and Thorogreen varieties.
- 2. Thick-seeded Baby Potato such as Baby Potato, Baby Fordhook, and Evergreen. Thick-seeded, such as Fordhook variety.

## IV. FROZEN BEANS, SPECKLED BUTTER (LIMA)

Frozen speckled butter (lima) beans are the frozen product prepared from the clean, sound, freshly-vined (but not seed-dry) seed of the speckled butter (lima) bean plant (*Phaseolus limensis*). The skins of the seed are pigmented, and the external colors range from a variegated speckling of green, pink, red, and/or lavender to purple. The product is prepared by shelling the pods; by washing, blanching, and properly draining the seeds that have been sorted and blended or otherwise prepared in accordance with good commercial practice. They are frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation of the product.

## V. FROZEN BROCCOLI

Frozen broccoli is the product prepared from the fresh, clean, sound stalks or shoots of the broccoli plant [*Brassica oleracea* (Italica group)] by trimming, washing, blanching, sorting, and properly draining. The product is frozen in accordance with good commercial practice and maintained at temperatures necessary for its preservation.

## A. Styles

- 1. Spears or stalks are the head and adjoining portions of the stem, with or without attached leaves, which may range in length from 9 cm (3.5 in.) to 15 cm (5.9 in.). The spears or stalks may be cut longitudinally.
- 2. Short spears or florets are the head and adjoining portions of the stem, with or without attached leaves, which may range in length from 2.5 cm (1 in.) to 9 cm (3.5 in.). Each short spear or floret must weigh more than 6 g (0.2 oz). The short spears or florets may be cut longitudinally.
- 3. Cut spears or short spears are cut into portions that may range in length from 2 cm (0.8 in.) to 5 cm (2 in.). Head material should be at least 62.5 g (2.2 oz) per 250 g (8.8 oz), and leaf material should not be more than 62.5 g (2.2 oz) per 250 g (8.8 oz).
- 4. Chopped spears or short spears are cut into portions that are less than 2 cm (0.8 in.) in length. Head material should be at least 12.5 g (0.4 oz) per 50 g (1.8 oz), and leaf material should not be more than 12.5 g (0.4 oz) per 50 g (1.8 oz).
- 5. Pieces or random cut pieces are cut or chopped portions of spears or short spears or other units that do not meet the requirements for cut or chopped styles.

#### VI. FROZEN BRUSSELS SPROUTS

Frozen brussels sprouts are the frozen product prepared from the clean, sound succulent heads of the brussels sprouts plant (*Brassica oleracea* L. var. gemmifera) by trimming, washing, blanching, and properly draining. The product is frozen in accordance with good commercial practice and maintained at temperatures necessary for its preservation.

## **VII. FROZEN CARROTS**

Frozen carrots are the clean and sound product prepared from the fresh root of the carrot plant (*Daucus carota*) by washing, sorting, peeling, trimming, and blanching; they are frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation of the product.

#### A. Styles

Wholes (or whole carrots) retain the approximate form of a whole carrot.

Halves or halved carrots are cut longitudinally into two units.

Quarters or quartered carrots are cut longitudinally into four approximately equal units. Carrots cut longitudinally or cut longitudinally and crosswise into six or eight units approximating the size and appearance of quartered carrots are also permitted in this style.

Slices or sliced carrots are sliced transversely to the longitudinal axis.

Diced carrots consist of approximately cube-shaped units.

Double-diced carrots consist of approximately rectangular shapes that resemble the equivalent of two cube-shaped units.

Strips are carrots that consists of approximate French-cut shapes, with flat-parallel or corrugated-parallel surfaces, one-half inch or more in length.

Chips are carrots that consist of predominantly small-sized units (such as less than one-half cube) and variously shaped pieces or slivers in which the longest-edge dimension approximates not more than one-half inch. Cut carrots consist of cut units that do not conform to any of the forgoing styles.

# VIII. FROZEN CAULIFLOWER

Frozen cauliflower is prepared from fresh flower heads of the cauliflower plant (*Brassica oleracea botrytis*) by trimming, washing, and blanching and is frozen and maintained at temperatures necessary for preservation of the product.

# A. Styles and Requirements

- 1. Clusters are individual segments of trimmed and cored cauliflower heads, which measure not less than 20 mm (0.75 in.) in the greatest dimension across the top of the unit. A maximum of 10% by weight of clusters less than 20 mm (0.75 in.) in the greatest dimension across the top of the unit are allowed.
- 2. Nuggets or small clusters are individual segments of trimmed and cored cauliflower heads, which measure from 6 mm (0.25 in.) to less than 20 mm (0.75 in.) in the greatest dimension across the top of the unit. A maximum of 20% by weight of clusters, 20 mm (0.75 in.) or greater, and a maximum of 10% by weight of clusters less than 6 mm in the greatest dimension across the top of the unit are allowed.

#### IX. FROZEN CORN ON THE COB

Frozen corn on the cob is the product prepared from sound, properly matured, fresh, sweet corn ears by removing husk and silk and by sorting, trimming, and washing to assure a clean and wholesome product. The ears are blanched and then frozen and stored at temperatures necessary for the preservation of the product.

#### A. Styles

- 1. Trimmed. Ears trimmed at both ends to remove tip and stalk ends and/or cut to specific lengths.
- 2. Natural. Ears trimmed at the stalk end only to remove all or most of the stalk.

#### B. Lengths

- 1. Regular. Ears that are predominantly over  $3\frac{1}{2}$  inches in length.
- 2. Ears which are predominantly  $3\frac{1}{2}$  inches or less in length.

Colors of frozen corn on the cob: Golden (or yellow); white.

#### X. FROZEN LEAFY GREENS

Frozen leafy greens are the frozen product prepared from the clean, sound, succulent leaves and stems of any one of the plants listed below by sorting, trimming, washing, blanching, and properly draining. The product is processed by freezing and maintained at temperatures necessary for its preservation. Any functional, optional ingredient(s) permissible under the law may be used to acidify and/or season the product.

# A. Types

Beet greens

Collards

Dandelion greens

Endive

Kale

Mustard greens

Spinach

Swiss chard

Turnip greens

Any other "market accepted" leafy green

# B. Styles

- 1. Leaf consists substantially of the leaf, cut or uncut, with or without adjoining portion of the stem.
- 2. Chopped consists of the leaf with or without adjoining portion of the stem that has been cut into small pieces less than approximately 20 mm (0.78 in.) in the longest dimension but not comminuted to a pulp or a puree.
- 3. Pureed consists of the leaf with or without an adjoining portion of the stem that has been comminuted to a pulp or a puree.

#### XI. FROZEN OKRA

Frozen okra is the product prepared from the clean, sound, succulent, and edible fresh pods of the okra plant (*Hibiscus esculentus*) of the green variety. The product may or may not be trimmed, is properly prepared and properly processed, and is then frozen and stored at temperatures necessary for preservation.

#### A. Styles

- 1. Whole okra consists of trimmed or untrimmed whole pods of any length that may possess an edible portion of the cap. The length of a whole pod is determined by measuring from the outermost point of the tip end of the pod to the outermost point of the stem end of the pod, exclusive of any inedible stem portion that may be present.
- 2. Cut okra is trimmed or untrimmed whole pods, which may possess an edible portion of cap, and which have been cut transversely into pieces of

approximately uniform length. The length of a unit of cut okra is determined by measuring the longitudinal axis of the unit.

#### XII. FROZEN ONION RINGS, BREADED, RAW, OR COOKED

Frozen breaded onion rings, hereinafter referred to as frozen onion rings, is the product prepared from clean and sound, fresh onion bulbs (*Allium cepa*) from which the root bases, tops, and outer skin have been removed. The onion bulbs are sliced and separated into rings, coated with batter (or breaded), and may or may not be deep fried in a suitable fat or oil bath. The product is prepared and frozen in accordance with good commercial practice and maintained at temperatures necessary for the proper preservation of the product.

# A. Types

The type of frozen onion rings applies to the method of preparation of the product, and includes

- 1. French fried onion rings that have been deep fried in a suitable fat or oil bath prior to freezing.
- 2. Raw breaded onion rings that have not been oil blanched or cooked prior to freezing.

# XIII. FROZEN PEAS

Frozen peas is the food in "package" form, prepared from the succulent seed of the pea plant of the species *Pisum sativum* L. Any suitable variety of pea may be used. It is blanched, drained, and preserved by freezing in such a way that the range of temperature of maximum crystallization is passed quickly. The freezing process should not be regarded as complete until the product temperature has reached  $-18^{\circ}$ C (0°F) or lower at the thermal center, after thermal stabilization. Such food may contain one, or any combination of two or more, of the following safe and suitable optional ingredients.

For more details see Chapter 26 and Appendix A.

#### XIV. FROZEN PEAS, FIELD AND BLACK-EYED

Frozen field peas and frozen black-eyed peas, hereafter referred to as frozen peas, are the frozen product prepared from clean, sound, fresh seed of proper maturity of the field pea plant (*Vigna sinensis*), by shelling, sorting, washing, blanching, and properly draining. The product is frozen and maintained at temperatures necessary for preservation. Frozen peas may contain succulent, unshelled pods (snaps) of the field pea plant or small-sieve round-type succulent pods of the green bean plant as an optional ingredient used as a garnish.

For more details see Chapter 26 and Appendix A.

#### XV. FROZEN PEPPERS, SWEET

Frozen sweet peppers are the frozen product prepared from fresh, clean, sound, firm pods of the common commercial varieties of sweet peppers, which have been properly prepared, may or may not be blanched, and are then frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation of the product.

# A. Types

Type I, green; Type II, red; Type III, mixed (green and red)

# B. Styles

- 1. Whole stemmed: whole unpeeled pepper pods with stem and core removed
- 2. Whole unstemmed: whole unpeeled pepper pods with stems trimmed to not more than  $\frac{1}{2}$  inch length
- 3. Halved: whole stemmed, unpeeled pepper pods that have been cut approximately in half from stem to blossom end
- 4. Sliced: whole stemmed, unpeeled pepper pods or pieces of pepper pods that have been cut into strips
- 5. Diced: whole stemmed, unpeeled pepper pods or pieces of pepper pods that have been cut into approximately square pieces measuring ½ inch or less
- 6. Unit: a whole unpeeled pepper pod or portion of a pepper pod in frozen sweet peppers

#### XVI. FROZEN POTATOES, FRENCH FRIED

Frozen French fried potatoes are prepared from mature, sound, white or Irish potatoes (*Solanum tuberosum*). The potatoes are washed, sorted, and trimmed as necessary to assure a clean and wholesome product. The potatoes may or may not be cut into pieces. The potatoes are processed in accordance with good commercial practice which includes deep frying or blanching in a suitable fat or oil and which may include the addition of any ingredient permissible under the law. The prepared product is frozen and is stored at temperatures necessary for its preservation.

#### A. Types

Frozen French fried potatoes are of two types, based principally on intended use, as follows:

- 1. Retail type. This type is intended for household consumption. It is normally packed in small packages that are labeled or marked for retail sales. It may be otherwise designated for such use.
- 2. Institutional type. This type is intended for the hotel, restaurant, or other large feeding establishment trade. Primary containers, usually 5 pounds or more, are often not as completely labeled as for retail sales.

#### B. Styles

#### 1. General

The style of frozen French fried potatoes is identified by the general size, shape, or other physical characteristics of the potato units. Styles with cut units may be further identified by substyles as follows:

- 1. Straight cut refers to smooth cut surfaces.
- 2. Crinkle cut refers to corrugated cut surfaces.

#### 2. Strips

This style consists of elongated pieces of potato with practically parallel sides and of any cross-sectional shape. This style may be further identified by the approximate dimensions of the cross-section, for example:

$$\frac{1}{4} \times \frac{1}{4}$$
 inch  
 $\frac{3}{8} \times \frac{3}{8}$  inch  
 $\frac{1}{2} \times \frac{1}{4}$  inch  
 $\frac{3}{8} \times \frac{3}{8}$  inch

Shoestring refers to a strip, either straight cut or crinkle cut, with a cross-section predominantly less than that of a square measuring  $\frac{3}{8} \times \frac{3}{8}$  inch.

#### 3. Slices

This style consists of pieces of potato with two practically parallel sides, and which otherwise conform generally to the shape of the potato. This style may also contain a normal amount of outside slices.

#### 4. Dices

This style consists of pieces of potato cut into approximate cubes.

#### 5. Rissolé

This style consists of whole or nearly whole potatoes.

Any other individually frozen French fried potato product may be designated as to style by a description of the size, shape, or other characteristic that differentiates it from the other styles.

#### C. Length Designations

#### 1. General

The length designations described in this section apply to strip styles only.

#### 2. Criteria for Length Designations of a Sample Unit

Frozen French fried potato strips are designated as to length in accordance with the following criteria. Percent, as used in this section, means the percentage, by count, of all strips of potato that are  $\frac{1}{2}$  inch in length or longer.

- 1. Extra long. Eighty (80)% or more are 2 inches in length or longer; and 30% or more are 3 inches in length or longer.
- 2. Long. Seventy (70)% or more are 2 inches in length or longer; and 15% or more are 3 inches in length or longer.
- 3. Medium. Fifty (50)% or more are 2 inches in length or longer.
- 4. Short. Less than 50% are 2 inches in length or longer.

# XVII. FROZEN POTATOES, HASH BROWNED

Frozen hash browned potatoes are prepared from mature, sound, white or Irish potatoes (*Solanum tuberosum*) that are washed, peeled, sorted, and trimmed to assure a clean and wholesome product. The potatoes so prepared are blanched, may or may not be fried, and are shredded or diced or chopped and frozen and stored at temperatures necessary for their preservation.

#### A. Styles

- 1. Shredded. Shredded potatoes are cut into thin strips with cross-sectional dimensions from 1 mm by 2 mm to 4 mm by 6 mm and formed into a solid mass before freezing.
- 2. Diced. Diced potatoes are cut into an approximately cube shape from 6 mm to 15 mm on an edge and loose frozen. They contain not more than 90 grams, per sample unit, of units smaller than one-half the volume of the predominant size unit.
- 3. Chopped. Chopped potatoes are random cut pieces predominantly less than 32 mm in their greatest dimension and loose frozen.

#### XVIII. FROZEN VEGETABLES, MIXED

Frozen mixed vegetables consist of three or more succulent vegetables, properly prepared and properly blanched; may contain vegetables (such as small pieces of sweet red peppers or sweet green peppers) added as garnish; and are frozen and maintained at temperatures necessary for the preservation of the product.

#### A. Kinds and Styles of Basic Vegetables

It is recommended that frozen mixed vegetables, other than small pieces of vegetables added as garnish, consist of the following kinds and styles of vegetables as basic vegetables:

- 1. Beans, green or wax: Cut styles, predominantly of  $\frac{1}{2}$  inch to  $\frac{1}{2}$  inch cuts
- 2. Beans, lima: Any single varietal type
- 3. Carrots: Diced style, predominantly of  $\frac{3}{8}$  inch to  $\frac{1}{2}$  inch cubes
- 4. Corn, sweet: Golden (or yellow) in whole kernel style
- 5. Peas: early type or sweet type

# **B.** Recommended Proportions of Ingredients

It is recommended that frozen mixed vegetables consist of three, four, or five basic vegetables in the following proportions:

- 1. Three vegetables. A mixture of three basic vegetables in which any one vegetable is not more than 40% by weight of all the frozen mixed vegetables.
- 2. Four vegetables. A mixture of four basic vegetables in which none of the vegetables is less than 8% by weight nor more than 35% by weight of all the frozen mixed vegetables.
- 3. Five vegetables. A mixture of five basic vegetables in which none of the vegetables is less than 8% by weight nor more than 30% by weight of all the frozen mixed vegetables.

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# **Quality Control in Frozen Vegetables**

Domingo Martínez-Romero, Salvador Castillo, and Daniel Valero Miguel Hernandez University, Orihuela, Spain

#### I. INTRODUCTION

Freezing is an effective mean of preservation that maintains the quality of foods almost to fresh product. Although freezing is one of the easiest and least time-consuming methods, it is not as economical as canning; but it retains more nutrients in the food if properly done. Most vegetables retain their natural color, flavor, and texture better when frozen than if other methods of food preservation are used. Natural enzymes in foods cause changes in the above parameters, and freezing delays this activity, though it does not stop it. Thus, to prevent further enzyme activity, vegetables need to be blanched in boiling water or steamed for a brief period of time before freezing. However, nutrient loss occurs during blanching, and these losses are greater than those from enzymatic activity if vegetables are not blanched. An alternative method is the addition of antioxidants, such as ascorbic acid. Freezing does not destroy spoilage organisms, such as bacteria, molds, and yeasts; it merely retards their growth temporarily. Once the food is thawed, microorganisms may continue to grow. On the other hand, the blanching process can destroy several microorganisms, especially the mesophiles. During the storage of frozen vegetables, moisture evaporation can render them dry and tough, with the development of off-flavors. To solve this problem, two options are available: provide high relative humidity throughout the storage period; and/or use moisture vapor-proof or resistant packaging.

Although freezing has the disadvantage of the initial investment for equipment for the food industry, the beneficial effects of the use of frozen vegetables in terms of their quality attributes will be higher. This chapter focuses on the physical, structural, nutritional, and sensorial changes during the freezing and frozen storage processes.

#### II. IMPORTANCE OF FROZEN VEGETABLES IN THE FOOD INDUSTRY

Among the "mild" or "new" technologies of minimal processing in foods, industrial freezing is undoubtedly the most satisfactory method of preserving quality during longer storage periods (1). Vegetables were found to be more palatable and have better color when frozen than when canned, while dehydrated vegetables were shown to be as good or better than the canned. In terms of energy use, cost, and product quality, freezing requires the shortest processing time. Although more energy is required to process and store vegetables by freezing than by canning or dehydration, the overall cost, including

packaging and cost of equipment, for preservation by freezing can be kept as low or lower than the cost for other methods of preservation.

The depletion of the ozone layer in the atmosphere caused by the use of chlorofluorocarbons is a leading concern for the global environment. This, together with high cost and high energy consumption, opens new challenges to the scientists and engineers of food freezing equipment, in terms of improved finished product quality, reduced processing costs, improved safety and environmental factors, and most importantly, consumer acceptance. In order to achieve the desired freezing results, many factors are involved in the freezing process that determine final product quality, such as freezing methods, product ice crystallization, freezer burn, freezing rate, packaging, and moisture losses (2).

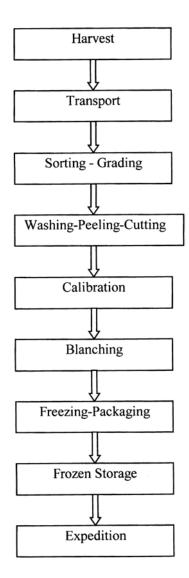
As nearly as Quick Frozen Foods International can determine, frozen food consumption in 13 European countries reached 11.1 million tons in the year 2000. Total retail sales of frozen foods in the U.S. reached more than \$25 billion in 1999, up over one billion dollars from 1998 (USDA-NAAS Agricultural Statistics 1999). In 1999, manufacturers' food service sales of frozen foods in the U.S. totaled \$40.6 billion. Thus the consumption of frozen vegetables has increased by 20% during the last 20 years (3).

#### III. PROCESSING OF FROZEN VEGETABLES

The freezing process is dependent on the freezing rate, the heat transfer coefficient, and the amount of heat removed from the food product. The freezing process time depends on the freezing rate, the amount of heat removed, the packaging and freezing methods used, the initial and final temperature desired, the thickness, and the food ingredients. The International Institute of Refrigeration (IIF) defines the freezing rate as the difference between the initial and final temperature of the product divided by the freezing time (4). The amount of heat to be removed and the cooling rate depend on the food structure and chemical composition. The freezing systems used affect ice crystal formation; large ice crystals induce product damage, which could be reduced with increased freezing rate. Several numerical mathematical models have been reported that consider assumptions including the irregular shape, the chemical composition, the heat transfer coefficient, and the type of freezing media used (5,6). Industries generally accept the target temperature of  $-18^{\circ}$ C (0°F) at the thermal center of the product for an efficient freezing process.

Several operations are needed during freezing that vary with the types of vegetables and the methods used, but general preparation procedures are summarized in Fig. 1, including postharvest preparation, blanching, freezing, and storage. Woodroof (7) reported general guidelines for harvesting, handling, and storing vegetables before commercial processing. To get an optimum quality after thawing, proper selection and control of raw material, cultivar, and maturity stage are very important factors. Thus vegetables should be harvested when they reach the peak of quality. During processing, vegetables should be handled promptly to avoid mechanical damage. During sorting and grading, insect-infected vegetables are removed, and during washing, dust, dirt, and insects are removed as well. In several cases, additional operations are needed, such as peeling, trimming, and cutting.

The most important step for enzyme inactivation is blanching. These enzymes cause the formation of off-flavors and discoloration during storage at freezing temperatures. An additional effect of blanching is the reduction of the number of microorganisms. There are several tests that can be used to assure that the blanching process has been adequately



**Figure 1** General flow diagram for processing of frozen vegetables.

performed, the most commonly used being peroxidase, catalase, lipoxygenase, and polyphenoloxidase. A high correlation has been established between development of off-flavors during freezing storage and remaining peroxidase and lipoxygenase activities, suggesting the imperative use of blanching to inactivate these enzymes in frozen vegetables for a better final quality (8). However, there are some reports that several vegetables, such as tomatoes, green peppers, celery, and mushrooms, can be frozen for up to 12 months at  $-18^{\circ}$ C without previous blanching and with no quality deterioration (9, 10).

Classically, the peroxidase level activity has been used to monitor quality changes in frozen vegetables, since increases in peroxidase are thought to indicate changes in flavor, color, and texture in vegetables. Thus measurement of peroxidase is often performed prior to blanching as a reference for determining the effectiveness of the blanching process (11), for which a 95% loss of enzyme activity following blanching is considered adequate (9). Still, several studies have established that residual lipoxygenase activity was closely related to off-flavor of leguminous vegetables (12, 13), while residual peroxidase activity has little effect on quality of frozen vegetables (14, 15). Increased peroxidase activity during frozen storage was found in peas blanched at 93–100°C for 1 min (16). Recently, it has shown

that although changes in total peroxidase activity may not predict flavor changes, the presence or absence of certain peroxidase isozymes may be useful in predicting off-flavor development in specific frozen corn genotypes (17).

There are a large number of published reports that reveal that the freezing rate is a key factor that preserves food products when physicochemical changes are studied. Quick freezing can be achieved by increasing temperature gradient between freezing media and food. Thus conventional mechanical freezing methods, such as forced air, are considered slower than liquefied gases such as nitrogen or carbon dioxide, which have low boiling points and freeze faster. These gases are commonly called "cryogenic gases," since their temperature range is cryogenic and the process is called cryogenic freezing. This type of freezing improves product quality and offers many advantages over mechanical freezing. Its benefits include reduction in freezing time (extremely fast), reduction in moisture and flavor losses, reduction in ice crystal formation, minimum product cell damage, and high heat transfer. It is also a flexible and versatile system.

# IV. PHYSICAL, STRUCTURAL, NUTRITIONAL, AND SENSORIAL CHANGES DURING FREEZING OF VEGETABLES

Processing operations destroy the cytoplasmatic structure, producing loss of turgor, weakness of cell wall, and some degree of cell separation. These changes have important effects on the texture of the vegetable, which is one of the most important quality factors of frozen vegetables from the consumer point of view. Quality frozen vegetables are directly correlated to pectin substances, firmness, texture, and histological structures.

The freezing temperature is the most critical factor affecting the cell structure in vegetables such as carrots (18). The blanched carrots reduced only 21% of their initial firmness, while in the raw samples this decrease was about 50%, the effect being due to the formation of a gel from the interaction between heat and pectic substances. In turn, a reduction in pectin extraction was observed (19). These results would confirm that damages occur in the middle lamella of the cells, the main damage being due to freezing rather than blanching. On the histological level, frozen raw samples showed physical changes, such as cell walls irregular in shape and separation among the cell layers, which were explained by the ice crystal effect. Contrarily, the cells of the blanched samples did not show tissue disruption.

As previously reported, freezing is an effective method of preservation, but comprehensive studies on physicochemical changes of foods during freezing and frozen storage have revealed that the freezing rate influences the quality of frozen and thawed vegetables. Thus cryogenic freezing could cause internal stress buildup leading to cracking or shattering that is critical and reversible in frozen materials (20). This mechanical damage is mainly due to both contraction and expansion of the volumetric changes associated with the water–ice transition (21). Physical properties, such as porosity and density, may also be affected by an ultrahigh freezing rate. Porosity indicates the amount of void space inside the vegetable, and a larger void space increases the possibility of internal stress. Density is usually proportional to the moisture content and inversely proportional to porosity; thus the greater the density, the higher the probabilities that stress will occur.

Water makes up over 90% of the weight of most produce and is held within the cell walls to give support, structure, and texture to the vegetable. Actually, the freezing of vegetables consists of freezing the water contained in the plant cell. When the water

freezes, it expands, and the ice crystals cause the cell walls to rupture. Freezing as quickly as possible can control the structural changes (cell wall disruption, internal stress, cracking, etc.). In rapid freezing, a large number of small ice crystal are formed, and less cell wall rupture can be expected by comparison with the formation of large ice crystals.

The size of the ice crystals affecting cell walls is related to the final quality after thawing. When a product is thawed, it is much softer than the raw product before frozen storage. The most typical example is tomato, which after being frozen and thawed turns liquid. The same can be concluded for celery and lettuce, which are not usually frozen. Textural changes due to freezing are not as apparent in products that are cooked before eating, since cooking also softens cell walls.

In frozen peppers, the maximum firmness was attributed to the activity of pectinmethylesterase (PME). Peppers blanched at 69°C showed increases in firmness, since PME was activated, generating free carboxylic acid groups that could cross-link with divalent cations. On the other hand, peppers blanched at 96°C showed inactivation of PME (22). Thus, for better texture attributes of this commodity, a decrease in temperature and an increase in time should be taken into account. In this sense, the use of calcium in combination with a low-temperature blanch is usually performed to maintain firmness during vegetable processing (23), through stimulation of the PME present in the cell walls by low-temperature blanching (24). The effects of this pretreatment on vegetables have been reported for canning (25), drying (26), and freezing (27).

The health benefits of vegetables are well recognized by nutritionists, but usually intakes are below recommendations. There are published reports linking fruit and vegetable intakes with a reduced risk of chronic diseases, such as cardiovascular disease and cancer (28). Among the nutrients, vitamins are essentials for human nutrition, and those acting as antioxidants deserve special attention. Vitamins C, vitamin A, and its precursor  $\beta$ -carotene are considered the main agents responsible for the protective effects because of their antioxidant and antiradical properties. Vegetables are estimated to provide 30% of the vitamin C and 20% of the vitamin A (as carotenes). The expansion of the frozen food industry has meant that most food that can be frozen is available for consumption throughout the year. This is especially important for vegetables that are dense in the essential nutrients such as vitamins. Obviously, we must give consideration to the fact that the vitamin C content varies according to other factors such as cultivation, processing, and storage conditions. Since vitamin C has a high solubility in water and a high sensitivity to heat, its content gives a good indicator of the quality and freshness of the frozen product (29). Since vitamin C is vulnerable to chemical and enzymatic oxidation, it is an appropriate marker for monitoring quality change during transportation, processing, and storage (30). During the freezing process, water-soluble substances are lost, especially during blanching (31). Thus in broccoli half of the vitamin C was lost after a blanching time of 60 s (?) before freezing in a fluid bed tunnel (32). Similarly, in fiddlehead greens, losses of vitamin C ranged from 30 to 38% as a result of freezing (33), and losses were also attributed to the blanching process. However, losses of vitamin C during the canning process are much higher (47–57%), since vitamin loss is partly dependent upon heating time and temperature.

In a comparative study of the vitamin C content of fresh and frozen vegetables (peas, beans, broccoli, carrots, and spinach), the author concluded that the vitamin C level in the commercial quick-frozen product is equal to or better than that in the fresh produce market and much better than that in the supermarket stored at fresh or ambient temperature. Also, the loss of ascorbic acid from all these vegetables is most probably dominated by enzyme-induced oxidation. The variation in the rate of loss demonstrates

the differing vulnerabilities of the different vegetables, such as surface area and mechanical damage, and their differing enzyme activities (30).

Carotenes are precursors of vitamin A, which is considered an essential nutrient for maintaining human health, but carotenes are susceptible to oxidation. The degradation of carotenes is associated with the development of off-flavors (34). Steam blanching is thought to result in little or no loss in  $\beta$ -carotene content (35). Similarly, the carotene retention was relatively high in frozen fiddlehead, since provitamin compounds are not very water-soluble (33). In a comparative study of carotene retention in carrots, broccoli, and spinach, the mean carotene content of the three vegetables decreased with time after thawing, but no differences were found for extended thawing time (36). This author also concludes that frozen and thawed vegetables exposed to home environmental conditions for 4 hours before cooking may not lose much carotene, whereas dehydration of vegetables may adversely affect carotene content.

#### V. CHANGES DURING STORAGE OF FROZEN VEGETABLES

Most vegetables will maintain high quality for 12 to 18 months at  $-18^{\circ}$ C. However, it is well known that during frozen storage the number of ice crystals will be reduced, while their size will increase. These changes are affected by fluctuations in storage temperature, which in turn can cause the migration of water vapor from the product to the surface of the container. The increase of ice crystals during prolonged frozen storage induces drip loss. Also, physical and chemical changes can be expected, which were recently summarized (37). Since at frozen storage temperatures, no microorganism proliferation can be expected, the loss of quality is mainly due to physical, chemical, and sensorial changes of higher magnitude than those detected during the freezing process.

The main physical changes of vegetable products during frozen storage are due to recrystallization and sublimation phenomena related to the ice crystals' stabilization inside the product and on the outside surface. Both phenomena are thought to be controlled by temperature. The recrystallization rate decreases at low temperatures, with no ice crystal growth at lower temperatures than  $-20^{\circ}$ C. The ice sublimation occurs in unwrapped vegetable products during temperature fluctuations during frozen storage, which causes product dehydration and accelerates the oxidative changes on the product surface area (38).

With respect to chemical changes, these are a consequence of the residual enzymatic action that produces loss of nutrients and color, and the occurrence of off-flavors. In terms of loss of nutrients, only small changes in carbohydrates may occur during frozen storage, as biochemical processes are delayed at freezing temperatures, but a reduction in water-soluble carbohydrates may occur as a result of water loss during thawing. In several vegetables, such as fiddlehead greens (33) and sweet potatoes (39), the nutritional parameters did not change throughout frozen storage. Minerals (Ca, K, Mg, and P) remained unchanged during 10 months of frozen storage, while sugars (fructose, glucose, and sucrose) showed increases during 9 months of storage, mainly due to starch being converted to sugars by reactivation of the enzymes involved.

Several vegetables, such as spinach, contain high concentrations of galactolipids and phospholipids among their fat-soluble components, which are used as substrates for lipid-acyl hydrolases such as galactolipases and phospholipases. The highly active thermal stability of these enzymes should be taken into account and the enzymes used as indicator enzymes for determining the quality deterioration during frozen storage (40). In this

vegetable, after 10 months of frozen storage 80% of the total folacin activity was retained with proper blanching and freezing processes (41).

The main factor determining the shelf life of frozen vegetables in prolonged storage is effective blanching, but several vegetables do not need blanching for optimum quality, as has been reported before. Unblanched vegetables, such as onions and leeks, were more acceptable after 15 months of storage than blanched samples (10), the lower quality being due mainly to loss of volatile oils during the blanching process.

In terms of the acceptability of vegetables, one of the most important quality factors is texture. Texture has even been associated as a criterion for the selection of raw materials. During frozen storage of asparagus an increase of the maximum force during cutting is produced, mainly owing to increased fiber content that affects the fibrous attributes by a lignification process either enzymatically or otherwise (42). The enzymatic lignification has been attributed to residual peroxidase activity after blanching at the basal and medium zones of the asparagus, but not in the apical ones. During the freezing (with Freon-12 immersion) and frozen storage of peas, changes in texture properties have been reported. Thus freezing at a higher rate resulted in smaller ice crystals and less structural damage. In terms of chewiness, increased values during storage were observed due to the dehydration effects (43). Moisture loss by evaporation of water on the surface area of a product produces freezer burn, a grainy brownish spot where the tissues become dry and tough. This surface freeze-dried area is very likely to develop off flavors. Moisture-proof wrap is used to prevent freezer burn.

In a recent study of carrots (44), pronounced differences in textural quality were found between the freezing method and frozen storage. Thus decreasing the temperature from  $-30^{\circ}$ C to  $-70^{\circ}$ C resulted in increasing maximum firmness, with no differences after 1 and 5 months of frozen storage.

With respect to color, frozen vegetables show alterations in natural pigments, such as chlorophyll, anthocyanins, and carotenoids, or enzymatic browning. Chlorophylls a and b have been shown to be the main compounds responsible for the green color of vegetables (45). Degradation of chlorophylls has been studied because their bright green color is usually more pleasing to the consumer than the brownish color of pheophytin a and b, which is a chemical conversion (46). Since chlorophyll in green tissues may depend on the nature of its association with lipoproteins of the chloroplast, the lipid peroxidation, as a consequence of being frozen, will be increased by the lipoxygenase action (47). Thus chlorophylls a and b were slightly degraded (about 16%) in frozen spinach, but small amounts of pheophytins a and b were detected, because the spinach had been blanched.

Anthocyanins are hydrosoluble pigments responsible for the red color of some vegetables. Under several conditions, they may be destroyed as a consequence of polyphenol enzymatic oxidation. The final result of this oxidation is the occurrence of enzymatic browning in frozen vegetables such as cauliflower, potato, and mushroom. This reaction is catalyzed by the enzyme polyphenoloxidase in the presence of oxygen and the production of quinines, which in turn can oxidize other substrates like ascorbic acid and anthocyanins. The most convenient parameter for monitoring enzymatic browning is related to CIE Lab. L\*, a\*, and b\* coordinates represent the color space, in which L\* indicates lightness, and a\* and b\* are the chromaticity coordinates. These parameters are expressed as positive or negative values. In the color space, + a is the red direction and – a is the green direction. Similarly, + b is the yellow direction and – b is the blue direction. In this sense, the enzymatic browning in potatoes has been correlated with decreases in parameter b\* (39).

The high or low acceptance of a specific frozen vegetable depends on its sensory attributes. Aroma and flavor together with color and texture are the most important. The lack of flavor and the absence of aroma are mainly due to the action of oxygen in the air on frozen product, producing rancid oxidative flavors. This can be solved with adequate wrapping material that does not permit air to pass into the vegetable, or by removing as much air as possible from the freezer bag or container before freezing.

#### VI. CONCLUSION

Freezing is a common process for long-term preservation of vegetables and is one of the best methods available in the food industry. Freezing retains the quality of vegetables near their fresh state, but interest has grown concerning the quality and shelf life of frozen vegetables. Consumption of frozen vegetables has increased by 20% during the last 20 years. However, during frozen storage, physical, chemical, and nutritional changes usually occur. To minimize these effects, blanching has been used traditionally in vegetable processing to slow quality deterioration caused by enzyme activity. Some benefits of blanching prior to freezing are color stability, reduced vitamin losses, texture improvement, and removal of undesirable substances.

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# 24

# Production, Freezing, and Storage of Tomato Sauces and Slices

Sheryl A. Barringer

The Ohio State University, Columbus, Ohio, U.S.A.

#### I. INTRODUCTION

# A. History

The tomato (*Lycopersicon esculentum*) is a member of the Solanaceae family. This family contains a number of plants important as human food, including potatoes, eggplant, and bell peppers. The name *Lycopersicon* derives from the Greek for wolf peach. The plant received its name from Anguillara and Marinello in 1561 (1) who mistakenly thought this name had already been given to the plant by Galen (2). Since Claudius Galen was referring to a plant growing in northern Africa in the second century A.D., 1400 years before the tomato arrived in that half of the world, he could hardly have been referring to the tomato. Another mistake was made when the genus was mistakenly spelled *Lycopersicum*. The misspelling appears to have been started by Hill (3) in 1773 and continued until it was pointed out by Druce (4) in 1914. This misspelling can still be seen in several places in the literature today. The first good description of the various tomato species, including the one commonly eaten today, was made by Miller in 1768 (5), hence texts frequently refer to the species as *L. esculentum* Mill (6).

Evidence points to the tomato originating in Central and South America. Although it is impossible to say for certain, from the large diversity of varieties grown in Mexico, its uses in native cooking, and the abundance of native names for the fruit, it appears that the original domestication took place in Mexico (2). The tomato was introduced into southern Europe soon after the discovery of the New World by Columbus. The Italian herbalist Pier Andrea Mattioli described a pomi d'oro (golden apple) plant bearing a golden fruit in 1544. Ten years later, a second edition mentioned a variety of the plant bearing red fruit. The diagram is clearly the typical tomato we eat today (2). In Britain and the United States the plant was originally used for medicinal and decorative purposes before finally becoming common as a food item in the mid eighteenth century. In 1893, the U.S. Supreme Court settled the fruit vs. vegetable question by ruling that the tomato was legally a vegetable for reasons of commerce.

# B. Biology

The tomato is a perennial plant industrially grown as an annual. Early selective breeding increased the size of the fruit, removed the ruffles, and decreased the seed content. The technique of back crossing varieties with desirable traits into existing varieties is widely used to increase pest and disease resistance and improve color, viscosity, and solids content. Genetic engineering has been used to improve the species further. Breeders continue to breed for improved yield, color, soluble solids, and machine harvestability (7).

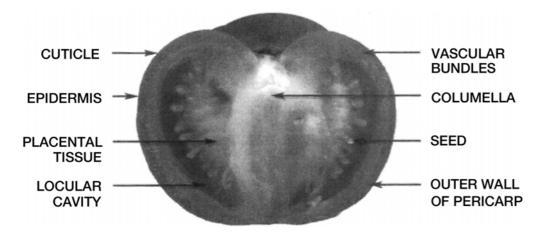
The portion of the tomato that is commonly eaten is the fruit, which is a berry. This fleshy berry consists of a skin over the outer wall and inner radial walls of pericarp containing the locular contents (Fig. 1). The skin is composed of a cuticle over the epidermis. The cuticle contains cuticular acids and waxes which make the fruit resistant to disease and insect attack, but they also make it difficult for processors to peel by steam or lye. Varieties have been bred to peel easily by the incorporation of the easy peel gene, but this is typically linked to worse insect and disease resistance. These varieties are also prone to cracking.

Just underneath the peel is attractive red flesh, rich in lycopene. If the fruit is overpeeled, this area is lost, exposing the less attractive yellowish vascular bundles. The interior locular cavities contain the seeds imbedded in a jellylike parenchyma. If these locular cavities are punctured, the interior will leak out, which is undesirable in whole peeled tomatoes.

# C. Growing and Harvesting

The tomato thrives in a wide range of latitudes, soil types, temperatures, and methods of cultivation, though it does best in soil with good drainage (7). Plants may be started as seedlings and planted when they are 1 to 2 months old, or they may be directly seeded in the field (7). Both methods are in commercial practice. It normally takes 4 months from the emergence of the seedling to harvest. In suitable climactic conditions, the growing season for tomatoes can be 300 days a year.

Fruit set is based on the night temperature. The optimum temperature is 59–68°F (15–20°C), and it fails to set at 55°F (13°C) or below (8,9). Fruit set is not sensitive to day length and will occur under day lengths varying from 7 to 19 hours. When there are high



**Figure 1** Cross section of a tomato.

levels of nitrogen in the soil, the plant will grow vigorously but not set fruit. The fruit requires 40 to 60 days from flowering to reach full ripeness (7).

The fruit of the tomato is climacteric. Unlike nonclimacteric fruit, the tomato produces ethylene when exposed to low concentrations of ethylene. In response to this gas, respiration increases and ripening occurs suddenly. For this reason, in years when the farmers need to force the field to ripen all at once, they spray their fields with a compound such as Ethrel that causes the tomatoes to produce ethylene.

Development of machine harvesting in the United States occurred in the 1950s and 1960s (7, 10). Mechanical harvesters cut the vines and carry them into the machine. The fruit are shaken off the vines and the vines returned to the field. The fruit falls onto conveyor belts where they are manually sorted to remove green and rotten fruit. The rotten and green fruit are returned to the fields. The advent of mechanical harvesting required a focus on breeding the plants for mechanical harvesting. The two critical factors were that the vines be jointless and that all of the fruit ripen at the same time. With mechanical harvesting, the vines of the older varieties would break off at the joints, leaving a stem on the fruit. Since machine harvesting requires that the entire field be harvested at once, concentrated fruit set and ripening are critical. In the United States, all processed tomatoes are machine harvested.

Characteristics of the tomato fruit must also be suitable for mechanical harvesting. Besides ripening uniformly, they must be able to withstand handling by a mechanical harvester and bulk hauling. Processing tomatoes are usually smaller than for the fresh market, have a high solids content, and possess a firm texture. They are typically roma varieties, which are pear shaped.

#### D. Production Statistics

The tomato is an important product both for domestic and export markets. The largest producer of tomatoes in the world is China, followed by the United States, Italy, and

 Table 1
 2000 World Production Statistics for Tomatoes

Location	Area harvested (1000 ha)	Production (1000 metric ton)
World	3635	99,125
Asia	1842	42,461
Europe	710	21,914
North and Central America	331	16,921
Africa	591	11,424
South America	151	5918
Oceania	9	486
China	754	18,347
United States	195	13,255
Italy	130	7091
Turkey	158	6600
Egypt	180	5950
India	360	5450

Source: Adapted from Ref. 11.

 Table 2
 2000 U.S. Production Statistics of Tomatoes for Processing

State	Harvested area (acres)	Production (tons)
California	271,000	10,286,500
Indiana	6600	229,020
Ohio	5400	158,710
Michigan	2800	84,000
Pennsylvania	1400	42,560
Other	2400	57,450

Source: Adapted from Ref. 12.

Turkey (Table 1). In the United States, the largest tomato producing state is California, followed by Indiana, Ohio, and Michigan (Table 2). Approximately 85% of the crop by weight is used for processing, making it the second largest vegetable crop for processing, by dollar value in the United States. About 45% of the world supply of processed tomato products is from California alone.

In the United States, the yield per acre continues to increase every year owing to improvement in varieties and growing practices. Over the last 20 years in California, breeding has resulted in a 1.54%/year increase in yield, no change in soluble solids, and a 1.15%/year improvement in color (13). An additional 1.16%/year improvement in yield occurred due to improvements in growing practices. The lack of improvement in soluble solids is likely because of the tradeoff between improving solids and improving yield.

#### E. Nutritional Value

The composition of the tomato is greatly affected by the variety, state of ripeness, year, climatic growing conditions, light, temperature, soil, fertilization, and irrigation. Tomato total solids vary from 5 to 10% (6) with 6% being average. Approximately half of the solids is reducing sugars, with slightly more fructose than glucose. Sucrose concentration is unimportant in tomatoes and rarely exceeds 0.1%. A quarter of the total solids consists of citric, malic, and dicarboxylic amino acids, lipids, and minerals. The remaining quarter, which can be separated as alcohol-insoluble solids, contains proteins, pectic substances, cellulose, and hemicellulose.

Tomatoes are mostly water (94%), a disadvantage when condensing the product to paste. They are a reasonably good source of vitamins C and A. In 1972 tomatoes provided 12.2% of the recommended daily allowance of vitamin C; only oranges and potatoes contributed more to the American diet (14). Tomatoes provided 9.5% of the vitamin A, second only to carrots. When major fruit and vegetable crops were ranked on the basis of their content of ten vitamins and minerals, the tomato occupied 16th place (7). However, when the amount that is consumed is taken into consideration, the tomato places first in its nutritional contribution to the American diet. This is because the tomato is a popular food, added to a wide variety of soup, meat, and pasta dishes.

# II. PROCESSING STEPS: TOMATOES TO JUICE, PASTE, AND FROZEN SAUCE

The majority of processed tomatoes are made into juice, which is condensed into paste. The paste is remanufactured into a wide variety of sauce products. The majority of frozen tomato products are sauces that are part of frozen meat and pasta entrees.

After harvesting, tomatoes are transported to the processing plant as soon as possible. Once at the plant, they are processed immediately, and if this is not possible they are stored in the shade. Fruit quality deteriorates rapidly while waiting to be processed. To unload the tomatoes, the gondolas are filled with water from overhead nozzles. Gates along the sides or undersides of the gondolas are opened, allowing the tomatoes to flow out into water flumes.

# A. Grading

The first step the tomatoes go through is to be graded to determine the price paid to the farmer. This is done at the processing facility or at a centralized station before going to the processing facility. Individual companies may set their own grading standards, use the voluntary USDA grading standards, or use locally determined standards, such as those of the Processing Tomato Advisory Board in California. The farmer is paid based on the percentage of tomatoes in each category. Typically companies hire USDA graders or hold an annual grading school to train their own graders.

The USDA divides tomatoes for processing into categories A, B, C, and culls (15). Grading is done on the basis of color and percentage of defects. Color can be determined visually to determine the percentage of the surface that is red, or with an electronic colorimeter on a composite raw juice sample. Defects include worms, worm damage, freeze damage, stems, mechanical damage, anthracnose, mold, and decay. The allowable percentage of extraneous matter may also be specified. Extraneous matter includes stems, vines, dirt, stones, and trash.

The Processing Tomato Advisory Board inspects all tomatoes for processing in California. Their standards are similar to those of the USDA but more geared for the paste industry. They inspect fruit for color, soluble solids, and damage (16). A sample of raw tomatoes is comminuted for color and soluble solids. The minimum tomato color instrument reading is 39. Soluble solids are reported for informational purposes. A load of tomatoes may be rejected for any of the following reasons: >2% of fruit is affected by worm or insect damage, >8% is affected by mold, >4% is green, or >3% contains material other than tomatoes, such as extraneous material, dirt, and detached stems.

Proper sampling procedure is essential to grade the lot accurately. The Processing Tomato Advisory Board requires that each sample from a bin or bulk load contain 50 pounds of tomatoes. Approximately one-half of the number of bins in the sample must be located below the top layer of the load. For bulk loads, containing 30 tons or less, two samples must be taken from the load.

#### B. Washing

Washing is a critical control step in producing tomato products with a low microbial count. A thorough washing removes dirt, mold, insects, drosophila eggs, and other contaminants. The efficiency of the washing process will determine microbial counts in the juice or paste (17, 18). Several methods can be used to increase the efficiency of the

washing step. Agitation increases the efficiency of soil removal. The warmer the water spray or dip, up to 194°F (90°C), the lower the microbial count (19, 20), though this is not typically done because of economic concerns. Lye or surfactants may be added to the water to improve the efficiency of dirt removal, although surfactants have been shown to promote infiltration of some bacteria into the tomato fruit by reducing the surface tension at the pores (21). The washing step also serves to cool the fruit. Since tomatoes are typically harvested on hot summer days, washing removes the field heat, slowing respiration and therefore quality loss.

Tomatoes are typically transported in a water flume to minimize damage to the fruit. Therefore tomato washing can be a separate step in a water tank, or built into the flume system. A water tank also serves to separate stones from the fruit, since the stones settle to the bottom. The final rinse step uses pressurized spray nozzles at the end of the soaking process. Flume water may be either recirculated or used in a counterflow system, so that the final rinse is with fresh water while the initial wash is done with used water. In either system, the first flume frequently inoculates rather than washes the tomatoes because all of the dirt in the truck is washed into the flume water (18). When the water is reused, high microbial counts on the fruit may result if careful controls are not kept.

Chlorine is frequently added to the water. Chlorine will not significantly reduce spores on the tomato itself because the residence time is too short. However, chlorine is effective at keeping down the number of spores present in the flume water (18). When there is a large amount of organic material in the water, such as occurs in dirty water, chlorine is used up rapidly, so it must be continuously monitored.

During fluming to the next step, upright stakes may be placed at intervals within the flume. Vines and leaves that have made it this far in the process are caught on the stakes. Periodically workers remove the trapped vines.

# C. Sorting

A series of sorters are used in a plant. The first sorter that is used, especially in small plants, is an inclined belt. The tomatoes are offloaded onto the belt. The round fruit rolls down the belt and into a water flume. The leaves, sticks, stones, and rotten tomatoes are carried up by the belt and dropped into a disposal bin.

Photoelectric color sorters are used in almost every plant to remove the green and pink tomatoes. These sorters work by allowing the tomatoes to fall between conveyor belts in front of the sensor. Unacceptable tomatoes are ejected by a pneumatic finger. A small percentage of green tomatoes in tomato juice does not adversely affect the quality. Green tomatoes bring down the pH but do not affect the color of the final product. In addition, less mature tomatoes result in a higher viscosity paste (22, 23). Pink or breaker tomatoes are a problem, however, because they decrease the redness of the juice.

The final sorting step is done by human sorters, who are more sensitive than mechanical sorters. Employees remove extraneous materials and rotten tomatoes from sorting tables. Sorting conveyors should require the employees to reach no more than 20" and move no more than 25'/min; the conveyors should consist of roller conveyors that turn the tomatoes as they travel, exposing all sides to the inspectors (24).

#### D. Break

The tomatoes are next put through a break system to be chopped. Some break systems operate under vacuum to minimize oxidation. In an industrial plant operating under

vacuum, no degradation of ascorbic acid occurs during the break process (25). When vacuum is not used, the higher the break temperature, the greater the loss of ascorbic acid (26).

Tomatoes can be processed into juice by either a hot break or a cold break method. In the hot break method, tomatoes are chopped and heated rapidly to at least 180°F (82°C) to inactivate the pectolytic enzymes polygalacturonase (PG) and pectin methylesterase (PME). Inactivation of these enzymes helps to maintain the maximum viscosity. Most juice is made by the hot break method, since most juice is concentrated to paste, and high viscosity is important in tomato paste used to make other products. Most hot break processes occur at 200–210°F (93–99°C).

In the cold break process, tomatoes are chopped and then mildly heated to accelerate enzymatic activity and increase the yield. Pectolytic enzyme activity is at a maximum at 140–151°F (60–66°C). Cold break juice has less destruction of color and flavor but also has a lower viscosity because of the activity of the enzymes. This juice can be made into paste, but its lower viscosity is a special advantage in tomato juice and juice-based drinks. In practice, both hot and cold break paste can be purchased with excellent color and high viscosity.

#### E. Extraction

After the break system, the comminuted tomatoes are put through an extractor, pulper, or finisher to remove the seeds and skins. Extraction of the juice is done with either a screw type or a paddle type extractor. Screw type extractors press the tomatoes between the screw and the screen. The screw is continually expanding along its length, forcing the tomato pulp through the screen. Very little air is incorporated into the juice, unlike in paddle type extractors, which beat the tomato against the screen, incorporating air. Air incorporation during extraction should be minimized because it oxidizes both lycopene and ascorbic acid. The screen size determines the finish, or particle size, which will affect the viscosity and texture.

#### F. Deaeration

Deaeration is frequently the next step to remove dissolved air incorporated during breaking or extraction. The juice is deaerated by pulling a vacuum as soon as possible because oxidation occurs rapidly at high temperatures. Deaeration also prevents foaming during concentration. If the product is not deaerated, substantial loss of vitamin C will occur.

#### G. Homogenization

The juice is homogenized to increase product viscosity and minimize serum separation. The homogenizer is similar to what is used for milk and other dairy products. The juice is forced through a narrow orifice at high pressure, shredding the suspended solids.

#### H. Concentration into Paste

The juice is next concentrated to paste. Concentration occurs in forced-circulation, multiple-effect vacuum evaporators. Typically three- or four-effect evaporators are used; most modern equipment now uses four effects. The temperature is raised as the juice goes

to each successive effect. A typical range is 118 to 180°F (48 to 82°C). Vapor is collected from later effects and used to heat the product in previous effects, conserving energy. The reduced pressure lowers the temperature, minimizing color and flavor loss.

The paste is concentrated to a final solids content of at least 24% NTSS (natural tomato soluble solids) to meet the USDA definition of paste. Commercial paste is available in a range of solids contents, finishes, and Bostwick consistencies. Common commercial concentrations are 26 and 31% NTSS. Typical finishes include 0.027" (0.69 mm), 0.033" (0.84 mm), 0.045" (1.1 mm), 0.060" (1.5 mm), 0.078" (2.0 mm) and 0.156" (4.0 mm). The larger the screen size, the coarser the particles and the larger the finish. Bostwick may range from 2.5 to 8 cm (tested at 12% NTSS).

# I. Aseptic Processing

The paste is heated in a tube-in-tube or scraped surface heat exchanger, held for a few minutes to pasteurize the product, then cooled and filled into sterile containers in an aseptic filler. A typical process might heat to 228°F (109°C) and then hold 2.25 minutes, or heat at 205°F (96°C) for 3 minutes. Aseptically processed products must be cooled before filling both to maintain high quality and because many aseptic packages will not withstand temperatures above 100°F (38°C). An aseptic bag-in-drum or bag-in-crate filler is used to fill the paste into previously steam sterilized bags. Paste is typically sold in 55 gallon drums or 300 gallon bag-in-boxes.

# J. Remanufacturing into Sauce and Freezing

Manufacturers of convenience meals buy tomato paste and remanufacture it by mixing it with water, particulates, and spices to create the desired sauce. Some sauce is made directly from fresh tomatoes during the tomato season, but this is less common. Sauce production from paste is more economical because it can be done during the off season using the equipment in tomato processing plants that would otherwise be unused. It is also cheaper to ship paste than sauce.

The sauce may be aseptically packaged and shipped to another plant, or immediately filled into the final container. Since the product will be frozen, it does not need to be retorted to make it shelf stable. Therefore, depending on the other ingredients, the product may not undergo any further heat processing. Once the sauce and other ingredients have been filled into the final container, the container is frozen on a spiral blast freezer at -30 to  $-40^{\circ}$ F (-34 to  $-40^{\circ}$ C).

#### K. Wastewater

Wastewater disposal is a critical issue in some locations, and it can put a tomato processor out of business owing to the high cost of disposal. One solution is to use flocculation to separate out the solids. The solids can then be disposed of as fertilizer on fields. The biological oxygen demand (BOD) of the remaining wastewater is low enough to permit economical disposal in the sewer system. Flocculation can be done with the addition of coagulants such as ferric chloride (27) or by pressurizing the sample, causing the water to absorb air. When the pressure is released, air bubbles are formed to attach to the solid waste, carrying it to the surface where it can be removed. Another solution is to pump wastewater, with or without the solids, directly into the fields to be used for irrigation.

#### III. PROCESSING: FROZEN SLICED TOMATOES

Currently, there are no frozen tomatoes available on the U.S. market. In Italy frozen tomatoes are successfully processed (28). Tomatoes are washed, sorted, blanched, peeled, sliced, diced, or left whole, inspected, and frozen on an IQF belt freezer. The whole peeled tomatoes are fluidized and quickly crust frozen in the first zone. The product is finish frozen on a second belt to  $0^{\circ}F$  ( $-18^{\circ}C$ ). A similar product was developed but not marketed in the United States. The tomatoes were sliced, blanched and cryogenically frozen. The company reported that the product remained firm, but it had to be stored below  $0^{\circ}F$  ( $-18^{\circ}C$ ) and was too expensive for the market.

# IV. QUALITY AND HOW IT IS AFFECTED BY GROWING AND PROCESSING

# A. Color and Lycopene

There are several methods for measuring color. The voluntary USDA grading standards for tomatoes to be processed use the Munsell Disk colorimeter (15). The Munsell Disk colorimeter consists of two spinning disks containing various percentages of red, yellow, black and gray. As the disks spin they visually combine to produce the same color as the tomato. USDA color comparators are plastic color standards which can be used to grade tomatoes visually. With fresh tomatoes, the Agtron colorimeter is common, especially for tomato juice and halves. The Agtron is an abridged spectrophotometer that measures the reflection at one to three wavelengths and reports the result as a color score. For processed tomato products, the Hunter colorimeter is common. The Hunter measures the L, a, and b values. The a and b values are put into a formula dependent on the machine, to correlate to color standards provided by UC Davis (29). The Agtron and Gardner can also be converted to these color scores. In the scientific literature, the L, a, b values are converted to hue angle (arc tangent b/a).

Consumers associate a red, dark-colored tomato product with good quality. The red color of tomatoes is created by the linear carotenoid lycopene. Lycopene is 80–90% of the carotenoids present. With the onset of ripening, the lycopene content increases (6, 7). The final lycopene concentration in the tomato depends on both the variety and the growing conditions. Some tomato varieties have been bred to be very high in lycopene, resulting in a bright red color. During growth, both light level and temperature affect the lycopene content. The effect of light on lycopene content is debated. Some authors report that shading increases lycopene content (30), while others report mixed results (31). The effect of temperature is much more straightforward. At high temperatures, over 86°F (30°C), lycopene does not develop (30, 32, 33).

Lycopene does not have any vitamin activity, but it may act as an antioxidant when consumed (34). A review of epidemiological studies found that the evidence for tomato products was strongest for the prevention of prostate, lung, and stomach cancer, with the possible prevention of pancreas, colon and rectum, esophagus, oral cavity, breast, and cervix cancer (35). The consumption of fresh tomatoes, tomato sauce, and pizza has been found to be significantly related to a lower incidence of prostate cancer, with tomato sauce having the strongest correlation (36). Since anticancer correlations are typically stronger to processed tomatoes than fresh tomatoes, several studies have looked at the effect of processing on lycopene. Tomato juice and paste have more bioavailable (absorbed into the blood) lycopene than fresh tomatoes when both are consumed with corn oil (34, 37). This

may be because of thermally induced rupture of cell walls and weakening of lycopeneprotein complexes releasing the lycopene, or improved extraction of lycopene into the lipophilic corn oil.

Color loss is accelerated by high temperature and exposure to oxygen during processing. The main cause of lycopene degradation is oxidation. Oxidation is complex and depends on many factors, including processing conditions, moisture, temperature, and the presence of pro- or antioxidants. Several processing steps are known to promote the oxidation of lycopene. During hot break, the hotter the break temperature, the greater the loss of color, even when operating under a vacuum (25). However in some varieties the break temperature affected color while in others it did not (38). The use of fine screens in juice extraction enhances oxidation because of the large surface area exposed to air and metal (39). Similarly, concentrating tomato juice to paste in the presence of oxygen degrades lycopene. It has been reported that heat concentration of tomato pulp can result in up to 57% loss of lycopene (40). However, other authors have reported that lycopene is very heat resistant and no changes occur during heat treatment (41). With current evaporators it is likely that very little destruction of lycopene occurs.

Processing also affects color owing to the formation of brown pigments. This is not necessarily detrimental, because a small amount of thermal damage resulting in a darker serum color increases the overall red appearance of tomato paste (42). Browning is caused by a number of reactions. Excessive heat treatment can cause browning owing to caramelization of the sugars. Amadori products, representing the onset of the Maillard reaction, occur during all stages of processing, including breaking, concentrating, and canning (43), although in the production of tomato paste the Maillard reaction is of minor importance (43). Degradation of ascorbic acid has been suggested to be the major cause of browning (44). Browning can be decreased by processing and storage at lower temperatures, by decreasing the pH to 2.5, and by the addition of sulfites (45).

#### B. Viscosity and Consistency

For liquid tomato products, viscosity is a very important quality parameter. It is second only to color as a measure of quality. Viscosity also has economic implications because the higher the viscosity of the tomato paste, the less paste needs to be added to reach the desired final product consistency. To the scientist, viscosity is determined by analytical rheometers, while consistency is an empirical measurement. To the consumer they are synonyms. Depending on the method, either the viscosity or the consistency of the product can be measured. Tomato products are non-Newtonian, and so many methods measure consistency rather than viscosity. The standard method for determining the consistency of most tomato products is the Bostwick consistometer. The Bostwick value is how far the material at 20°C flows under its own weight along a flat trough in 30 seconds. Tomato concentrates are typically measured at 12% NTSS to remove the effect of solids. Theoretically, this can be modeled as a slump flow (46). The Bostwick measures the shear stress under a fixed shear rate. Efflux viscometers such as the Libby tube (for tomato juice) and the Canon-Fenske (for serum viscosity) measure shear rate under fixed shear stress.

The viscosity of tomato products is determined by the solids content, serum viscosity, and physical characteristics of the cell wall material. The solids content is affected by the cultivar but is primarily determined by the degree of concentration. The serum viscosity is largely determined by the pectin. Pectin is a structural cell wall polysaccharide. The primary component of pectin is polygalacturonic acid, a homopolymer of (1–4) alpha-D-galacturonic acid and rhamnogalacturonans. Some of the

carboxyl groups are esterified with methyl alcohol. Pectin methylesterase (PME) removes these ester groups. This leaves the pectin vulnerable to attack by polygalacturonase (PG), which cleaves between the galacturonic acid rings in the middle of the pectin chain, greatly reducing the viscosity. During the break process heat is used to inactivate pectolytic enzymes, but these enzymes are released during crushing and act very quickly. Genetic modification has been used to produce plants with either an antisense PME (47) or a PG (48) gene to inactivate the enzyme, producing juice with a significantly higher viscosity. The physical state of the cell wall fragments affects viscosity by determining how easily the particles slide past each other. Most tomato products are homogenized to create more linear particles, which increases the viscosity.

Conditions during processing, such as temperature, screen size, and blade speed, will affect the final viscosity. Hot break juice is typically of a higher viscosity than cold break juice owing to inactivation of the enzymes that degrade pectin. At very high break temperatures, such as 212°F (100°C), the structure collapses and the viscosity decreases again (25), though this effect is not always observed (49). The screen size and blade speed during extraction are also important factors. The effect of screen size is not a simple relationship. A higher viscosity is produced using a screen size of 1.0 mm then either 0.5 mm or 1.5 mm (50). Other studies have found no effect of finisher size on final viscosity (25). The faster the blade, the higher the viscosity. The higher the evaporation temperature, the greater the loss of viscosity (25).

#### C. Serum Separation

Serum separation can be a significant problem in liquid tomato products. Serum separation occurs when the solids begin to settle out of the solution, leaving the clear straw-colored serum as a layer on top of the product. Preventing serum separation requires that the insoluble particles remain in a stable suspension throughout the serum. Generally, the higher the viscosity, the less serum separation occurs.

Factors that affect the quantity and quality of the solids determine the degree of serum separation that occurs. The higher the temperature during the break process, the less serum separation occurs (25). Hot break juice has less serum separation than cold break. This may be due to greater retention of intact pectin in the hot break juice (49), though Robinson et al. (51) found that the total amount of pectin did not affect the degree of settling in tomato juice. The cellulose fiber may be more important in preventing serum separation than the pectin (51, 52). Addition of pectinases degrades the pectin, increasing the dispersal of cellulose from the cell walls. The expansion of this cellulose minimizes serum separation (51).

Homogenization is commonly used to shred the cells, increasing the number of particles in solution and creating cells with ragged edges that reduce serum separation. The result is particles that will not efficiently pack and settle. Of these two effects, changing the shape of the particles is more important than their change in size (51). Evaporator temperature during concentration has little effect on serum separation (25).

#### D. Flavor

The flavor of tomatoes is determined by the variety used, its stage of ripeness, and the conditions of processing. Typically, varieties have not been bred for optimal flavor, though some work has focused on breeding tomatoes with improved flavor. Processing tomatoes are picked fully ripe, so the concern that tomatoes that are picked mature but

unripe have less flavor is not important. Processing generally causes a loss of flavor. Processes are not optimized for best flavor retention, but practices that maximize color usually also maximize flavor retention. When flavor is evaluated, it is done by sensory evaluation. Gas chromatography is used to determine the exact volatiles present.

Flavor is made up of taste and odor. The sweet-sour taste of tomatoes is due to their sugar and organic acid content. The most important of these are citric acid and fructose (53). The sugar/acid ratio is frequently used to rate the taste of tomatoes, though Stevens et al. (53) recommend against it because tomatoes with a higher concentration of both sugars and acids taste better than those with low concentrations, for the same ratio. The free amino acids, salts, and their buffers also affect the character and intensity of the taste (54). The odor of tomatoes is created by the over 400 volatiles that have been identified in tomato fruit (54, 55). No one single volatile is responsible for producing the characteristic tomato flavor. The volatiles that appear to be most important to fresh tomato flavor include *cis*-3-hexenal, 2-isobutylthiazole, beta ionone, hexenal, *trans*-2-hexenal, *cis*-3-hexenol, *trans*-2-trans-4-decadienal, 6-methyl-5-hepten-2-one, and 1-penten-3-one (54, 55).

Processed tomato products have a distinctively different aroma from fresh tomato products. This is due to both the loss and the creation of volatiles. Heating drives away many of the volatiles. Oxidative decomposition of carotenoids causes the formation of terpenes and terpenelike compounds. The Maillard reaction produces volatile carbonyl and sulfur compounds.

Many of the volatiles responsible for the fresh tomato flavor are lost during processing, especially *cis*-3-hexenal and hexenal (56). *Cis*-3-hexenal, an important component of fresh tomato flavor, is rapidly transformed into the more stable *trans*-2-hexenal, so it is not present in heat processed products (57). The amount of 2-isobutylthiazole, responsible for a tomato leaf green aroma, diminishes during the manufacture of tomato puree and paste (58).

Other volatiles are created. The breakdown of sugars and carotenoids produces compounds responsible for the cooked odor. Dimethyl sulfide is a major contributor to the aroma of heated tomato products (54,56,59–60). Its contribution to the characteristic flavor of canned tomato juice is more than 50% (60). Linalool (59), dimethyl trisulfide, 1-octen-3-one (61), acetaldehyde, and geranylacetone (57) may also contribute to the cooked aroma. Pyrrolidone carboxylic acid, which is formed during heat treatment, has been ascribed to an off-flavor that occasionally appears (62). This compound, formed by cyclization of glutamine, arises as early as during the break process (43).

Heating causes degradation of some flavor volatiles as well as inactivating lipoxygenase and associated enzymes, which are responsible for producing some of the characteristic fresh tomato flavor (63). However, hot break has been found to produce a better flavor by some authors (38) and a less fresh flavor by others (63). Within one study, the flavor of one variety may be rated better as cold break than hot break and another variety the reverse (26, 38). This may in part be because some panelists prefer the flavor of heat-treated tomato juice to fresh juice (60).

# E. pH and Titratable Acidity

The pH of tomatoes has been reported to range from 3.9 to 4.9, or in standard cultivars, 4.0 to 4.7 (64). The critical issue with tomatoes is to ensure that they have a pH below 4.7, so that they can be processed as high-acid foods. The lower the pH, the greater the inhibition of *Bacillus coagulans* and the less likely flat sour spoilage is to occur (65). Within the range of mature, red ripe to overly mature tomatoes, the more mature the tomato, the

higher the pH. Thus pH is more likely to be a concern at the end of the season. The USDA standards of identity allow organic acids to be added to lower the pH as needed during processing.

The acid content of tomatoes varies according to maturity, climatic conditions, and cultural method. The acid concentration is important because it affects the flavor and pH. Citric and malic are the most abundant acids. The malic acid contribution falls quickly as the fruit turns red, while the citric acid content is fairly stable (66). The average acidity of processing tomatoes is about 0.35% expressed as citric acid (55). The total acid content increases during ripening to the breaker stage and then decreases.

The relationship between total acidity and pH is not a simple inverse relationship. The phosphorous in the fruit acts as a buffer, regulating the pH. Of the environmental factors, the potassium content of the soil most strongly affects the total acid content of the fruit. The higher the potassium content, the greater the acidity.

Processing conditions further affect the pH and acidity of processed tomato products. During processing the pH decreases and the total acid content increases (67, 68), though the citric acid content may increase (67) or decrease (68). Hot break juice has a lower titratable acidity (70) and a higher pH than cold break juice (26, 49). The difference is caused by the pectolytic enzymes still present in the cold break juice breaking down the pectin (71).

# F. Total Solids, Degrees Brix, NTSS, and Sugar Content

Tomato solids are important because they affect the yield and consistency of the finished product. Due to the time required to make total solids measurements, soluble solids are more frequently measured. Soluble solids are measured with a refractometer, which measures the refractive index of the solution. The refractive index is dependent on the concentration and the temperature of solutes in the solution, so many refractometers are temperature controlled. The majority of the soluble solids are sugars, so refractometers are calibrated directly in percentage sugar, or "Brix. NTSS or natural tomato soluble solids are the same as "Brix, minus any added salt.

The sugar content reaches a peak in tomatoes when the fruit is fully ripe (66). Light probably has a more profound effect on sugar concentration in tomatoes than any other environmental factor (6). The seasonal trends in the sugar content of glass house grown tomatoes have been found to follow roughly the pattern of solar radiation (69). Even the minor shading provided by foliage reduces the total sugar content by up to 13% (31).

During heat treatment, the reducing sugar content decreases due to caramelization, the Maillard reaction, and the formation of 5-hydroxymethyl furfural. The amount of sugar lost depends on the process. Studies have reported as much as a 19% loss in processed tomato juice (67) and a 5% loss during spray drying (72).

# G. Enzymes

Pectin methylesterase and polygalacturonase break down the pectin chains, reducing the product viscosity as described in the section on viscosity. Lipoxygenase and associated enzymes cause lipid oxidation off-flavors during storage if the product is not adequately heat treated. Lipoxygenase and its associated enzymes are also responsible for the development of fresh tomato flavor during the cold break process.

#### V. QUALITY LOSS DURING FREEZING AND FROZEN STORAGE

Initial quality loss occurs during the freezing process. Liquid nitrogen frozen slices have a significantly better texture than slices frozen at  $-34^{\circ}$ C (73). Addition of calcium chloride has been shown to improve hedonic ratings of frozen tomato slices in some cases (74) but not in others (73). However, these slices were still given lower hedonic ratings than fresh slices (73, 74). The flavor of frozen slices was also found to be significantly worse than that of fresh slices (74).

Further quality loss occurs during storage. Most studies have focused on unblanched tomatoes. The enzymes remain active, resulting in significant losses in color, vitamin C, flavor, and texture. In unblanched dices at  $-4^{\circ}F$  ( $-20^{\circ}C$ ), the loss of color and carotenoids was modeled as a linear decrease with time (75). The loss is accelerated by oxygen and light and can be decreased by the addition of spices with antioxidant properties (76). Color changes are observable by sensory evaluation after 6 months at  $-4^{\circ}F$  ( $-20^{\circ}C$ ) or 12 months at  $-22^{\circ}F$  ( $-30^{\circ}C$ ) (77). After a year of storage, 43% of vitamin C is lost at  $-4^{\circ}F$  ( $-20^{\circ}C$ ) and 70% at  $-22^{\circ}F$  ( $-30^{\circ}C$ ) (77). Hedonic ratings of color, texture, and flavor deteriorate significantly (57, 77, 78).

However, thawed, unblanched dices were still organoleptically acceptable for use on pizza after 12 months at  $-22^{\circ}F$  ( $-30^{\circ}C$ ), or 9 months at  $-4^{\circ}F$  ( $-20^{\circ}C$ ). They were still acceptable for use in vegetable salads after 12 months at  $-22^{\circ}F$  ( $-30^{\circ}C$ ), or 6 months at  $-4^{\circ}F$  ( $-20^{\circ}C$ ) (77).

Blanching appears to stop quality loss during storage. No difference in vitamin C, or hedonic ratings for color, appearance, texture, flavor, or taste, were seen in slices after 6 weeks at  $0^{\circ}F$  (-18°C) (78). In puree after 4 months at  $0^{\circ}F$  (-18°C), a 25% loss of vitamin C has been reported, but the flavor was still judged to be acceptable (79).

During the first three months of storage at  $-4^{\circ}F$  ( $-20^{\circ}C$ ), there is a decrease in the number of microorganisms, including pathogens, on frozen tomato slices (80). However, the counts after 9 months of storage were still high enough to present a safety concern. Thus proper sanitation must be used during preparation, since little microbial death occurs during storage.

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# **25**

# Frozen French Fried Potatoes and Quality Assurance

#### Y. H. Hui

Science Technology System, West Sacramento, California, U.S.A.

#### I. INTRODUCTION

This chapter describes the general process of manufacturing frozen French fried potatoes and some aspects of quality assurance of the products. The information has been derived from grading and inspection documents issued by the U.S. Department of Agriculture (USDA).

#### II. PRODUCTS COVERED

The principal products covered are the traditional French fries, potatoes cut into strips, partially deep fried, and frozen. The standard also may be applied to any potato product, regardless of shape or composition if it is similarly processed and frozen. This includes products fabricated primarily from mashed, crushed, cut, or shredded potatoes and which are preformed into units prior to frying and freezing. Because of the difficulty of keeping oils in suitable condition, deep frying has not been popular with home cooks. With the discovery and development of frozen French fries, home consumption has increased rapidly. Institutional use also is increasing yearly. Some believe that yearly production now far exceeds that of any other frozen vegetable.

#### A. Areas of Production

The white potato is the world's most important vegetable crop. It is grown to some extent in all agricultural areas in the United States. Certain types of potatoes, particularly those of low solids contents, are not always suitable for manufacturing. Therefore extensive production of frozen French fried potatoes is limited principally to those areas where the raw product is most suitable. They are in general the Idaho, eastern Oregon, and Washington areas, the San Joaquin Valley of California, and the state of Maine. There is also some sizeable production in New Jersey, eastern Pennsylvania, Michigan, and the Red River Valley of Minnesota.

Because of varietal differences and growing conditions, potatoes from these widely separated areas have their own characteristics, particularly with respect to flavor and mealiness. Very mealy French fries are produced principally from the Russett varieties in the Pacific Northwest. In other sections of the country, the solids of the raw product are generally lower and the finished French fries have a slightly different flavor, they are less mealy than those from the Northwest region. These regional differences have given rise to claims of superiority of the product based principally on the degree of mealiness. This is partially a matter of personal preference. Good quality French fries are produced in all the leading producing areas.

#### **B.** Varieties

There are several dozen recognized market varieties of potatoes grown in the United States, and more are being developed each year. The Irish Cobbler is probably the most widely grown, and the Katahdin is grown in the greatest volume. Among the more popular varieties are the Russet Burbank (Idaho), Cobbler, Katahdin, White Rose, Green Mountain, Bliss, Triumph (red), Russett Rural, Kennebec, Norgold, and Pontiac. Various varieties of potatoes have their own cooking qualities. Some are more popular for one quality than for another, that is, bakers, boilers, and fryers. The characteristics of the various varieties are not distinct, and they are not always the same in all growing areas and all seasonal conditions. Therefore no one variety is used entirely for the production of frozen French fried potatoes.

# C. Receiving

Frozen French fries are usually produced fairly close to the source of supply but occasionally the raw product may be drawn from any of the principal potato producing areas of the country. At time of harvest, most late varieties of potatoes have a total sugar content of less than 1% of total solids. Such potatoes are usually suitable for manufacturing into frozen French fries. After the potatoes are stored for a period of time at 40°F or less the starch content partially changes to sugar, and the potatoes, if used immediately out of storage, may be unsatisfactory because of the high sugar content. Sugar in excess of 2–3% (based on dry potato weight) may render the potato practically worthless for deep frying. Such potatoes subjected to high temperatures develop black or brown areas, spots, or streaks, owing to carmalizing and burning of the sugar.

They also may have a burnt or sweet taste. Most potatoes can be "conditioned" by storing them for a period of time, at least two weeks, at near 60 or 70°F. In conditioning, the potato starts to respire, a process that uses the sugar and converts a portion of it back to starch. If the potatoes have been subjected to excessive cold storage, that is, down to 32 to 34°F or lower, trouble in conditioning may be encountered, such as tissue breakdown leading to rotting.

# D. Determining the Quality and Condition of Raw Potatoes for Frying Purposes

Processors try to evaluate and classify the quality of the raw product prior to purchase or processing. Two of the most important characteristics that indicate quality are specific gravity, closely associated with moisture content, and the degree to which starch has been converted to sugar. These will affect the texture and the color of the product. The size and shape of the potatoes is also important because of the cost of operations, the yield, the

length of the units, and the number of slivers and irregular-shaped pieces. The presence of off-odors and off-flavors such as those caused by some insecticides is at times very serious.

No entirely satisfactory method seems to have been developed to predetermine the cooking quality of potatoes. Specific gravity tests, which to some extent indicate the degree of mealiness, are sometimes made. Picric acid color tests may also be made. These indicate to some extent the relative amount of sugars present. The objectionable flavor of benzine hexachloride—an insecticide—can be detected by boiling and mashing a sample of the potatoes.

Probably the most satisfactory method of determining the quality of the raw product is to subject a representative sample of the lot to a cooking test similar to the process that will be used in manufacture. The USDA, in cooperation with the State of Maine and certain potato processors, has developed a series of color photographs that show various degrees of darkening after a standard fry. Comparison of actual samples of cooked potato to the photographs provides a fairly accurate means of evaluating the quality of a load of potatoes for the purpose of making French fries. Some large users base their raw potato contracts on the fry colors shown in the USDA Color Standards for Frozen French Fried Potatoes.

#### III. MANUFACTURE

Each processor of frozen French fried potatoes has his own particular methods of manufacture. However, there are a number of things common to all processors. The following outline describes the principal steps in manufacture. These steps may vary with different manufacturers. The principles given here are basic.

After receiving potatoes or having withdrawn them from storage bins or "conditioning" cellars, frying and/or suitable chemical tests are made from representative samples of the lot to determine whether the potatoes are in condition to be processed.

#### A. Washing

If the potatoes are in a condition suitable for processing, they are washed and may be run through hot water to remove some of the dirt and to loosen the peel. The potatoes may then be sized prior to peeling. Some plants flume the potatoes from the place of storage to the peelers, thus accomplishing the preliminary washing in this manner.

#### B. Peeling

After excessive dirt is washed off, the potatoes are dropped into peeling machines. These may be steam, lye, abrasive, or roller type peelers. Steam and lye peelers give a quick cook that loosens the skin or peel but does not penetrate deeply into the potatoes. The peelings are then removed by passing the potatoes through rubber rollers and water sprays. In abrasive, roller type peelers the skin or peel is removed without the addition of heat.

# C. Trimming

The potatoes after leaving the peeling machines are trimmed on wide moving belts. In the better plants, these belts are arranged in sections so that each potato is picked up by an operator, examined for defects, trimmed if necessary, and tossed over a barrier onto

another section of the belt. This procedure is much more satisfactory than trying to stir the potatoes on a single belt, because many potatoes many miss any examination at all on the single belt. At this time the potatoes may also be sorted for size; the larger ones go to institutional lines, the smaller ones into the retail and by-product lines.

In some plants, electric eye sorters are installed after the slicing operation to eliminate blemished units, thus cutting down on the amount of hand sorting and trimming of the whole potatoes.

### D. Slicing

After the potatoes are trimmed and sorted to size they go to the slicing machines. These slicers usually consist of two sets of knives, either rotary or fixed. One set of knives slices the potato to the desired thickness. The potato slices are then passed through another set of knives, which cut the slices to strips if desired. The size of the strips depends on the wishes of the management. It may vary from one quarter by one quarter inch to one half by one half inch in cross section. The usual size for retail sales is  $\frac{3}{8}$  by  $\frac{3}{8}$  inch. Poor slicing may be caused by small or irregular-shaped potatoes, by poor machinery, or by good machinery not properly used or adjusted. The knives may be straight or corrugated.

# E. Sizing

In the process of cutting potatoes into strips, there is always a certain amount of slivers and otherwise irregularly shaped pieces. A certain number of these pieces are expected in this product and are allowed for in the tolerances contained in the grade standards. It is usually necessary, however, to pass the cut potatoes over some type of shaker screen to remove a portion of the small pieces and slivers. The amount of chip material removed depends to some extent on the wishes of the purchaser. Processors do not like to remove any more than they have to because of the loss in yield.

# F. By-products

The excessive loss of potato material because of the peeling, trimming, and screening operations causes processors to consider by-products to utilize this material. Often this material is wasted; however, a large number of products, such as patties, puffs, and shreds, and diced and mashed potatoes, have been developed to utilize this material. Dehydrated flakes is also an important use. Where satisfactory use is made of screenings and sound throwouts, there is less tendency to keep this material in the frozen French fry pack.

#### G. Desugaring

Sugar in excessive amounts or irregular quantities of sugar between units may cause French fried potatoes to have dark or irregular color, poor texture, and/or unpleasant taste. Proper harvesting, good storage, and conditioning after storage helps in the control of the sugars. However, conditioning and storing potatoes is an expensive process and is avoided whenever possible. Reasonably satisfactory methods of rapid equalization of sugar content have been developed. The methods used vary between manufacturers. However, the basic principle is to run the sliced potatoes through a water bath leaching out a portion of the surface sugar and then replacing the sugar to the desired level by blanching in a sugar solution (partially cooking the product), so that upon frying the color

between units will be uniform. This method, based on a patented process, evens the surface sugar content between units. The sugar content of the whole slice is not greatly affected.

# H. Blanching

The sliced potatoes are usually run through a hot water blanch that partially cooks the product. This may or may not be a part of the desugaring process referred to previously. After blanching, the product may pass beneath heating units, under forced draft, which tends to remove most of the excessive moisture before the potato enters the fryer.

# I. Frying

Frying of the potatoes is usually a continuous process. The potatoes enter the hot oil on or under a draper-chain type belt traveling a certain distance before being removed, or an undulating type belt moves the potatoes in and out of the oil; the oil flow moves the potatoes along from one end of the fryer to the other. Some manufacturers use a double fry. That is, after the first fry, at approximately 350 to 370°F, the potatoes fall onto another belt and enter another fryer at about the same temperature. There are several reasons for this; the principal one being that there is more even coloring because of the stirring of the potatoes as they fall from one belt to another.

#### J. Fat or Oil

The term fat refers to a product that is plastic at room temperature such as lard or the usual vegetable shortenings. Oils are liquid at ordinary temperatures. The terms are here used to mean the same thing. Any animal or vegetable fat or oil that does not impart an unpleasant flavor to the French fries is suitable for the purpose. Different processors use different oils. Peanut oil, cottonseed oil, or mixtures of vegetable oils including some amount of soybean oil are used. Lard, which is hog fat, imparts a flavor to the French fries that is particularly desirable to some people. Soybean oil in large amounts may impart a flavor that is usually disliked. Hydrogenated lard is tasteless.

One of the biggest difficulties in proper frying is to maintain the fat or oil in good condition. Fats and oils deteriorate rapidly with the addition of water under high temperature, and also when in contact with bronze or brass fittings. When the frying oil deteriorates, it darkens in color and develops unpleasant odors that are imparted to the product. Dark bits of burnt carbon maybe deposited on the French fries, giving them an unpleasant appearance, Quality control people often use the amount of free fatty acid present in the oil as an indication of the degree of deterioration. A range in the area of 0.4 to 1.0% is regarded as normal.

Potatoes lose up to 30 to 40% of their weight, principally water, during frying. Water is removed from the oil by a partial vacuum created by the upward draft in the hood and attaching stack covering the frying vat. Condensation from the hood is carried away by troughs along the edge of the hood. The tendency to deteriorate may be checked by eliminating bronze or brass fittings, adjusting the size of the fryer to volume of potatoes, using oil that will stand the highest temperature in the system, and adding new oil from time to time.

In the better processing methods, the amount of oil used is very small, and it is usually heated by superheated steam in a heat exchanger rather than by direct flame. This

keeps the oil in all parts of the system well below the scorching point. Usually the oil is filtered continually to remove charred materials and is thus kept clean.

# K. Time and Temperature

There are many variants to be considered in determining the time and temperature of the fry. Potatoes of high specific gravity require less time to lose their excess moisture than those of low specific gravity. Different varieties of potatoes and potatoes in different conditions with respect to reducing sugars may require different cooks to attain a uniform degree of color. Certain markets seem to want potatoes fried much lighter in color than do other markets. French fries packed for institutional use, where an additional fry is to be given by the users, are usually fried to a much lighter color than are retail packs where the cooking is usually completed by the oven method. These light colored fries are usually designated as oil-blanched or par-fried.

Probably the most satisfactory means of arriving at the correct time and temperature for frying is to fry representative sample batches of each new load. If the samples come out too dark, either the time or the temperature, or both, of the cook may be reduced; if too light, they may be increased. In most plants, quality control people watch the color of the fries as they leave the fryer, both for overall color and for uniformity of color, and recommend suitable adjustments of the process. These recommendations may be based on experience or on actual color plates or models that are provided as guides for the operators. The USDA color standards may be used for this purpose. Immediately after coming from the fryer heat may be applied to drive off excess surface oil. In many plants the potatoes are cooled quickly after the fry by a blast of air. This air blast may be designed to blow off the outer oil that clings to the hot potatoes.

# L. Packaging

Packaging is usually accomplished by automatic machinery that places the proper weight of French fries into each package. The packages are usually weighed individually and adjusted for exact weight. This packaging operation may take place before freezing or, if belt freezing is used, after the potatoes emerge from the freezer. The resulting end product of the belt freezing method is easier to handle because the units separate easily, whereas the plate frozen product may emerge as one solid unit. Broken units are more common when the product is belt frozen.

#### IV. INSPECTION DURING PACKING OPERATIONS

The basic principles of in-plant inspection apply in general to inspection during manufacture. Processing operations as outlined above and as observed in the plant will suggest observations to be made and the best points to make them.

Good sanitation, particularly with respect to conveyors, belts, cutting machines, and machinery that comes in contact with cut potatoes is particularly important because yeasts, molds, and bacteria thrive in a potato-water medium and odors develop quickly. Also, there may be a buildup of oil or grease between fryer and packaging lines.

Samples checked for color at the discharge end of the fryers will indicate whether the potatoes are in proper condition for frying. Samples taken over the last shaker and just prior to packaging can be checked for defects (including defectives per pound). Cooking

tests should be made as soon as practical after freezing in order to develop all the information necessary for the in-plant inspection report.

#### V. INSPECTING THE PRODUCT

# A. Sample Unit Size

Any change in sample unit sizes from those specified in the standards changes the probability of the lot of passing or failing the intended grade. The size of the sample unit used is, therefore, very important. The sizes are

In the retail type, 16 ounces of product selected either from a production line or from one or more market packages.

In the institutional type, 32 ounces of product selected either from a production line or from one market package.

Caution: Make every effort to obtain a representative sample. French fries, particularly strip styles, tend to stratify themselves with vibration. Therefore try to take from the full depth on the belt or package rather than from the top. Often a sweep across the entire width of a belt would be better than from just one spot.

# B. Initial Fry Color, Types, Styles, and Length Designations

These items provide a much needed standardized language for trading, since these terms—previously widely used—were subject to much individual interpretation. Accurate identification of the fry color, type, style, and length designation is very important. They should be reported on all certificates.

#### 1. Fry Color

Color changes caused by frying require special consideration. Keep in mind the following definitions:

- Fry color refers to the color change that occurs in the potato units solely because of the initial frying or the oil-blanch process.
- Fry color of the individual units is ascertained by comparing them with the USDA Color Standards for Frozen French Fried Potatoes. The range of color includes the color space, up to but not including the next darkest color.
- Fry color of the sample unit is the range of colors that occur in the frozen product before any additional heating.
- Fry color designation of a sample unit is the fry color designation appropriate to the ranges specified in the Standards.
- The USDA Color Standards referenced are a series of colors that depict changes that occur solely because of the frying process. They are numbers 0, 1, 2, 3, and 4.

These designations are amplified as follows:

- USDA No. 0 in the color standards has no browning caused by frying. The background colors of all these illustrations is yellow. Background colors of potato strips are usually basically white. They may be creamy-white, yellow-white, or any other characteristic color. See Table 1.
- Refry color means the actual color of a potato unit after heating—either deep frying or in an oven.

**Table 1** USDA Colors

USDA Color	Optional fry color designation	Application to a sample unit
No. 0	Extra light	A sample unit may be designated Extra Light if almost all of the units have no fry color at the edges as in USDA No. 0.
No. 1	Light	A sample unit may be designated Light if most of the potato units are lighter than USDA Color No. 2.
No. 2	Medium light	A sample unit may be designated Medium Light if most of the potato units are lighter than USDA Color No. 3 but may include Color No. 1.
No. 3	Medium	A sample unit may be designated Medium if most of the potato units are darker than USDA Color No. 2 and may further range in color as dark as Color No. 4.
No. 4	Dark	A sample unit may be designated Dark if most of the potato units are darker than USDA Color No. 3. This designation may contain units similar to No. 4, and darker. Sample units designated No. 4 Dark fry color are not allowed in Grade A.

Refry color of the sample unit is the range of colors that are present after heating in preparation for grading.

Refry color designation is the color designation that may be given to the sample unit after heating. The appropriate criterion for this designation is given in Refry or (after heating) Color Range Guide in later discussion.

#### 2. Types

Many plants pack primarily for retail, and others primarily for the institutional market. Some pack an identical product for both types. For retail, however, the fry process usually has progressed to the extent that there is some color change and sufficient oil is retained that French fried potatoes of characteristic texture may be prepared by heating the product in an oven. For institutional use the units are usually processed very lightly, resulting in little color change and often not enough oil retention for proper preparation in an oven. This is often referred to as oil-blanched or par-fried.

The determination of type is based on intended use. You must make this determination on the information available to you.

Guidelines for this decision are as follows:

- 1. Small packages (5 pounds or less) which are labeled or marked as is customary or required for retail sales, and particularly those bearing official USDA marks, are considered to be of the retail type. Five-pound packages that are so marked, however, may be considered to be of the institutional type if declared by the applicant to be intended for such use.
- 2. Packages of any size that are not labeled or marked as is customary or required for retail sales and display are considered to be of the institutional type unless specifically declared to be retail by the applicant for inspection.
- 3. If the product is unpackaged, as on belts or in tote bins, or if the packaging does not indicate the intended use, it is considered to be retail type, and the retail type

defective allowances apply. Such a lot, however, may be considered to be institutional type if so requested by the applicant.

### 3. Styles

a. Strips. This style should be designated as Straight cutStraight cut-shoestringCrinkle cut

The cross-sectional dimensions of the strips are important to the buyer. Because of the nature of the product these are not very uniform. Designate the cross sections, therefore, as "approximate" and to  $\frac{1}{8}$  inch—as approximately  $\frac{5}{8} \times \frac{5}{8}$  inch, or  $\frac{5}{8} \times \frac{3}{4}$  inch, etc. The cross-sectional dimension of crinkle cut strips are normally measured from "hill" to "valley".

b. Slices, Dices, Rissolé, Other—See the chapter Frozen Vegetables and Product Description.

# 4. Length Designations (Applies Only to Strips)

Length in French fries is closely related to quality and value for many purposes. Extra long, for example, is usually considered a premium pack for institutional use. It is seldom packed for retail, since it present difficulties in packaging in retail-size containers and often requires sizing of the uncut potatoes. Long is packed in both retail and institutional types and is often considered a premium pack for retail. Medium is the usual retail size.

With the exception of short lengths, which are specifically excluded from U.S. Grade A, the length of units is not considered to be a factor of quality under the U.S. standards. Short lengths may, however, be designated U.S. Grade A Short if the strips meet the other requirements of U.S. Grade A.

The lengths designated in the standards are intended to provide workable and much needed definitions for terms that are regularly used in trading.

Determining the length. The length designation may be determined readily by isolating the strips that are 3 inches in length or longer and those that are less than 2 inches in length. The percentages of 2 inches in length or longer and 3 inches in length or longer can be readily calculated. Chips, slivers, pieces, and strips that are less than  $\frac{1}{2}$  inch in length are not considered in the total count.

See the USDA File Code 130-A-75 for the description and scale drawing of the Vegetable Strip Sizer, an effective device for sizing the strips.

The minimum equipment for inspecting frozen French fried potatoes is

- 1. Grading scale
- 2. Large flat trays
- 3. Ruler (size and length grading plate)
- 4. Percentage calculator
- 5. Authorized visual USDA Color Standards for Frozen French Fried Potatoes
- 6. Vegetable strip sizer
- 7. Oven of suitable type, or deep frying equipment

# C. Preparation of the Sample

The factors of color and defects are partially evaluated before the product is heated. Often when a package is opened there is a film of frost on the units which masks the color, or if storage conditions have not been good there may be a crust of ice or a heavy coating of ice crystals. If there is any appreciable condition of frost, ice crystals, or icing in the sample, thaw until the condition disappears to the extent that the color can be properly evaluated. Icing is usually not serious but the thawing of the sample in the oven may add enough moisture to the potatoes that they are soggy when cooked and also cause an explosion when put into hot frying oil (see Texture).

The sample should be examined for color designation using the USDA Color Standards as a guide, as discussed under color.

#### VI. QUALITY EVALUATION

#### A. Grade Factors that Are Not Scored

#### 1. Flavor

The flavor of French fried potatoes is affected by the conditions of the potatoes with respect to sugar or sunburn, by the condition of the fat or oil used, and, to a certain extent, by the variety of the potatoes, the type of soil, and climatic conditions; whether or not certain insecticides have been applied to the growing potatoes.

Good flavor is required in Grades A and A Short and at least reasonably good flavor in Grade B. Sweetness, bitterness, rancidity of oil, and pronounced scorched or caramelized flavor and odors are the usual reasons for lowering the evaluation of flavor from Good to only Reasonably good. Any definitely objectionable flavors or odors would be cause for lowering the grade of the product to Substandard. After the product has been heated in a suitable manner, taste it and smell it and classify its flavor as Good, Reasonably good, or Poor.

#### 2. Color Designation of a Sample Unit

The exact color of good quality potatoes varies considerably because of varietal differences, physical differences, types of fat used, areas of production, and other causes. It also varies because of the amount of color change induced by the frying process. These values are important to buyers because certain markets and certain important customers have strong preferences as to the lightness or darkness of the brown coloring.

Two separate and distinct color determinations are required:

- 1. Classifying the fry color of the sample unit as to its value (that is, its lightness or darkness) in order to establish the proper fry color designations
- 2. Evaluation and assigning the score points for color in compliance with the standards, giving consideration to color changes in the refried product.

Grade A, Good Color—27 to 30 points. This color is bright and typical of the product and meets the uniformity of fry color given for

No. 0—Extra Light

No. 1—Light

No. 2—Medium Light

No. 3—Medium

and meets the uniformity of refry color given in the Re-Fry Color Range Guide. Grade B, Reasonably Good Color—24 to 26 points (limiting rule). This color must be characteristic of French fried potatoes—not dull or off-color. It may exceed the fry

color variation given for any of the USDA colors—including No. 4—dark. After heating, the variation in the refry color may exceed those indicated in the guide but may not seriously detract from the appearance of the product.

Substandard—0 to 23 points (limiting rule). Lots that darken quickly—before the interiors are cooked—or very irregular would fall into this classification.

# B. Uniformity of Size and Symmetry

Uniformity of length of normal shaped strips is not considered under this factor. Consideration is given to the effect of any chips—as defined—on the appearance of the product and the percent by count of small pieces, slivers, and/or irregular pieces. In assigning score points be guided by the following:

#### Grade A

20 points—almost no chips, and/or

(Strips) no more than 5% of small pieces, slivers, and/or irregular pieces

(Other styles) almost perfect uniformity in size and shape of the units

18 points—chips present but not to materially detract from appearance, and/or

(Strips) more than 5% to 15% of small pieces, slivers, and/or irregular pieces.

(Other styles) high degree of uniformity in the size and shape of the units

19 points—by interpolation.

#### Grade B

17 points—chips present materially detract and/or

(Strips) more than 15% to 20% small pieces, slivers, and/or irregular pieces

(Other styles) reasonably uniform in size and shape

16 points—chips present that approach serious appearance, and/or

(Strips) more than 20% to 30% small pieces, slivers, and/or irregular pieces

(Other styles) variation in the size and shape of the units detracting noticeably from the appearance of the product

#### C. Defects

Defects are carefully defined in the standards as insignificant imperfections, minor defects, and major defects. Defectives are potato units affected with defects, as defined in the standards as minor defective or major defective. It is defectives rather than defects which are scored against.

#### 1. Considerations

For each grade, three separate types of deficiencies are considered. While the principal consideration is major and minor defectives, three factors must be considered in assigning the scores for the sample units:

- 1. The total effect of all faults that might be present, whether specifically mentioned. This is the "overall clause." Among such are extraneous materials, insignificant imperfections, and carbon specks or defects (as defined), and obnoxious blemishes that are much worse in appearance than the usual major defects.
- 2. The effect of any carbon specks on the appearance of the product.

3. The allowances for minor and major defectives as specified in Tables 2 and 3 of the standards.

#### 2. Defect Tables in the Standards

Defectives allowed in these tables are not averages. Sample units that fail the applicable requirement are allowable in the sample only as regular deviants.

# 3. Assigning the Score for Defects

- 1. Segregate the minor and major defectives in the sample unit and record them on the score sheet as (1) total (major and minor) and (2) major.
- 2. Assign a tentative score for defects as indicated by the following guide.
- 3. Adjust the score point if appropriate by giving consideration to the overall clause and the effect of any carbon specks present. This becomes the defect score for the sample unit.

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Table 2 Standards—All Styles Except Shoe Strings and Dices

			RETAIL TYPE	E					
Grade	de Point Defective Possible combinations of defecti								
	20	Total	0–3	_	_				
	20	Major	0	_	_				
	10	Total	4–5	1–3	_				
A	19	Major	0	1					
	10	Total	4–5	_	_				
	18	Major	1						
	1.77	Total	6–9	6–9	2-5				
D	17	Major	0	1	2				
В	1.6	Total	6–9	_	_				
	16	Major	2	_	_				
		INST	TITUTIONAL T	ГҮРЕ					
Grade	Point	Defective Possible combinations of defectives							
	20	Total	0–6	1–4	_				
	20	Major	0	1		_			
	10	Total	7–18	5–18	2-12	3-8			
A	19	Major	0	1	2	3			
	10	Total	13-18	9–18	4–18				
	18	Major	2	3	4				
	17	Total	19–28	5–23	6–18	_			
D	17	Major	0-4	5	6	_			
В	16	Total	24–28	19–28	7–28	_			

5

6

16

Major

Table 3 Standards—Shoestring, Strips, and Dices

RETAIL TYPE							
Grade	Point	Defective	efective Possible combinations of defect				
	20	Total Major	0–5 0	1–2 1	_		
A	19	Total Major	6–9 0	3–5 1	2–5 2		
	18	Total Major	6–9 2	6–9 1			
В	17	Total Major	10–18 0–2	3–15 3	4–8 4		
	16	Total Major	16–18 3	9–18 4	5–18 5		

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Grade	Point	Defective	Possi	ons of defectives		
	20	Total	0–10 0	1–8 1–2	_	
A 19	19	Major Total	11–28	9–28	3–21	5–18
11		Major Total	0 22–28	1–2 19–28	3–4 7–28	5–6
	18	Major Total	3–4 29–36	5–6 9–30	7–8	_
В	17	Major	0–8	9–10	_	_
	16	Total Major	31–36 9–12	11–36 11–12	_	_

Groups are inclusive, i.e.,  $\frac{3-5}{1}$  means  $\frac{3}{1}, \frac{4}{1}$ , or  $\frac{5}{1}$   $\frac{\text{Total}}{\text{Major}}$ .

Guide for assigning tentative score for defects—subject to adjustment for overall clause and for carbon specks.

#### D. Texture

Texture is evaluated within 3 minutes after heating the product as specified, and while it is well above room temperature.

#### E. Heating the Product

Oven method. The method of reheating specified in the standards is similar to that employed by the housewife. Crumpled foil is placed in the bottom of the pan in order to prevent excessive burning of the potatoes where they touch the metal pan. Fifteen minutes at 400°F is a minimum for most potatoes. The time depends on the size of the units, the sugar content, the type of oven (gas or electric), the number of samples in the oven, and how well it is ventilated. Trial runs are usually necessary to determine the proper time to

cook any lot of potatoes in the available equipment. Potatoes are properly cooked when the interior of the largest units has lost the raw potato taste. This method should be used when it is obvious that the product is intended for home use and that cooking directions call for the oven method. Exceptions may be made when test runs have shown that the deep fat method (below) gives results comparable to the oven method on the particular potatoes.

Deep fat method. Frozen French fried potatoes prepared for institutional use usually have a lighter fry color than those prepared for the retail trade. This is because the institutions using these potatoes will give them a short fry in oil. This additional fry can be adjusted in time and temperature so that the finished French fries will have the desired color. This desired color may be light or fairly dark depending upon the preference of the cooks. Also the directions on some retail packages provide for an additional cook in hot oil rather than an oven cook. For this reason, provision is made in the United States standards for heating the product by any other method that will give comparable results.

Deep fat frying is probably preferred for inspection use because of the speed with which the samples can be run. It should always be used where the product is light in color and/or obviously intended for institutional use. Where large numbers of samples are to be inspected, a deep fat fryer of the type marketed for household use and provided with an automatic heat control is very useful. If only an occasional sample is to be inspected, equally good results may be obtained by using a small stew pan with a wire dipper. With this equipment it is necessary to have an emersion thermometer capable of registering up to 600 degrees Fahrenheit. Also, new automatic frying pans can be obtained with heat control units.

Heat at least 100 units to determine the score for character. The temperature of the oil is very important. The temperature must be high during the entire refry time or the results will be in error. 100 units in a very large tank such as may be available for in-plant inspection would not lower the temperature significantly. With a quart or pint of oil only a few units can be fried at a time without lowering the oil temperature. Good texture varies somewhat with the varieties used and the area of production. It may vary from a somewhat cheeselike, very fine grained texture to a coarse-grained and almost powdery texture.

Usual variations from acceptable texture are

Sogginess. As the name implies, this refers to a wet pasty or mushy condition loaded with either water or oil. It may be a basic characteristic of the potatoes, or it may be induced by frying at too low a temperature. Often only a portion of the potato becomes soggy. Both the amount of the unit affected and the degree of sogginess must be considered in estimating the effect on texture. Score the unit only if 50% of its length (or less if very objectionable) is so affected.

Hardness. Interior portions that are very firm, sometimes oily to the touch, and raw in taste even if well cooked. Often, as with sogginess, only a portion of a strip or slice is hard. Score such units only if 50% (or less if very objectionable) of its length is so affected.

*Pull away*. Interior portion of a strip that has withdrawn from the outer shell, voiding  $\frac{1}{3}$  of the cross-sectional area of a regular strip or  $\frac{2}{3}$  of the cross section of a shoestring.

Crisp outer surface. Really crisp outer surfaces is a texture fault in any grade. A slight crispness is expected in Grade A and the surfaces may be slightly hard or slightly tough in Grade B. Keep in mind that excessive cooking will increase the crispness of the outer surfaces.

Sugary ends. A unit that has a dark and often soft rubbery end, caused by excess sugar.

Excessive oiliness. For reasons that are not always explainable, an unusual amount of oil is sometimes retained by the fries. It is very objectionable to buyers as it affects the texture adversely. Excessive oiliness can often be detected by the feel of the units prior to the heating. If excessive oiliness does not disappear with normal preparation, lower the texture score to reflect this condition.

# F. Score Points

The exact score points to assign requires careful preparation of the sample. Consider all the factors affecting texture and assign scores as indicated in the following guide:

Scoring procedure: heat 100 strips to determine the texture score. The number of points deducted from a possible 30 points will depend on the overall excellence of the sample. Consideration must also be given for those units in a sample that have a soggy or hard texture, or show pull away, or have excessively oily outer surfaces. Sugary ends not serious enough to be considered defects would fall into this category. The sample shall be practically free of such units to score in the Grade A range. Percentages ranging from 0% to 10% by count, depending on the seriousness of the defective units, are acceptable in this grade.

Prepared French fried potatoes that are scored 24 to 26 points for texture must be reasonably free from soggy or hard texture, pull away, or sugary ends, or those that do not have a crisp outer surface.

Score 26 points if there are 11 to 15% by count of these scorable units or if the units with slightly soggy or hard interior portions, or soft or slightly hard exterior surfaces, materially affect the overall appearance or eating quality of the product.

Score 25 points if there are 16 to 20% by count of the scoreable units and 24 points if there are 21 to 25% by count.

# **26**

# Frozen Peas: Standard and Grade

# **Peggy Stanfield**

Dietetic Resources, Twin Falls, Idaho, U.S.A.

In the United States, two federal agencies have the responsibility to ensure that the canned vegetables in the market are safe and do not pose any economic fraud. The U.S. Food and Drug Administration (FDA) issues regulations to achieve both goals. The U.S. Department of Agriculture (USDA) issues voluntary guidelines in addition to achieving the same goals, aiming at facilitating commerce. The information in this chapter has been modified from such regulations and guidelines.

#### I. STANDARDIZED FROZEN PEAS: FDA REQUIREMENTS

Appendix A of this volume reproduces the FDA requirements for standardized frozen peas.

# II. FROZEN FIELD PEAS AND FROZEN BLACK-EYED PEAS: USDA STANDARDS FOR GRADES AND GRADING METHODS

While the FDA establishes the requirements for standardized frozen peas to assure the safety of the product and to avoid economic fraud, the USDA develops grade standards to supplement the goals of the FDA, that is, to facilitate commerce between the sellers and buyers of frozen peas.

Voluntary U.S. grade standards are issued under the authority of the Agricultural Marketing Act of 1946, which provides for the development of official U.S. grades to designate different levels of quality. These grade standards are available for use by producers, suppliers, buyers, and consumers. As in the case of other standards for grades of processed fruits and vegetables, these standards are designed to facilitate orderly marketing by providing a convenient basis for buying and selling, for establishing quality control programs, and for determining loan values.

The standards also serve as a basis for the inspection and grading of commodities by the Federal Inspection Service, the only agency authorized to approve the designation of U.S. grades as referenced in the standards, as provided under the Agricultural Marketing Act of 1946. This service, available as on-line (in-plant) or lot inspection and grading of all

processed fruit and vegetable products, is offered to interested parties, upon application, on a fee-for-service basis. The verification of some specific recommendations, requirements, or tolerances contained in the standards can be accomplished only by the use of online inspection procedures. In all instances, a grade can be assigned based on final product factors or characteristics.

In addition to the U.S. grade standards, grading manuals or instructions for inspection of several processed fruits and vegetables are available upon request for a nominal fee. These manuals or instructions contain detailed interpretations of the grade standards and provide step-by-step procedures for grading the product.

Grade standards are issued by the Department after careful consideration of all data and views submitted, and the Department welcomes suggestions that might aid in improving the standards in future revisions.

This chapter presents the USDA voluntary grade standards for frozen field peas and frozen black-eyed peas. The coverage is as follows:

#### III. 7 CFR 52.1661 PRODUCT DESCRIPTION

Frozen field peas and frozen black-eyed peas, called frozen peas in these standards, mean the frozen product prepared from clean, sound, fresh seed of proper maturity of the field pea plant (*Vigna sinensis*), by shelling, sorting, washing, blanching, and properly draining. The product is frozen and maintained at temperatures necessary for preservation. Frozen peas may contain succulent, unshelled pods (snaps) of the field pea plant or small sieve round type succulent pods of the green bean plant as an optional ingredient used as a garnish.

#### IV. 7 CFR 52.1662. STYLES

- 1. Frozen peas
- 2. Frozen peas with snaps

#### V. 7 CFR 52.1663. TYPES

#### A. Single Type

Frozen peas that have distinct similarities of color and shape for the type are not considered "mixed." Single types include, but are not limited to, the following:

- 1. Black-eyed peas or other similar varietal types, such as purple-hull peas, that have a light colored skin, a definite eye (contrasting color around the hilum), and are bean shaped.
- 2. Crowder peas of various groups, such as Brown Crowder, that are nearly round in shape and have blunt or square ends.
- 3. Cream peas of various groups, including White Acre, that have a solid cream-colored skin and are generally bean shaped.
- 4. Field peas means any varietal group or type of the field pea plant that has similar color and shape characteristics and includes black-eye peas, Crowder peas, and Cream peas.

# B. Mixed Type

Frozen peas that are a mixture of two or more distinct single varietal groups or are not distinguishable as a single varietal group shall be considered to be of the mixed type.

#### VI. 7 CFR 52.1664. DEFINITIONS OF TERMS

# A. Acceptable Quality Level (AQL)

The maximum percent of defective units or the maximum number of defects per hundred units of product that, for the purpose of acceptance sampling, can be considered satisfactory as a process average.

# B. Appearance

The overall appearance of a sample unit refers to its brightness and uniformity. The color of snaps in the "frozen peas with snaps" style is considered under the overall appearance.

# 1. Good Appearance

The sample unit has a bright and uniform overall appearance.

# 2. Reasonably Good Appearance

The sample unit has an overall appearance that may be dull.

#### C. Blemished

Blemished means discolored, spotted, or damaged by any means to the extent that the appearance or eating quality is materially affected.

#### D. Broken

Broken means that the skin or portions of the skin, the cotyledon or portions of the cotyledon, have become separated from the unit. "Broken" is not applicable to snaps in the style of frozen peas with snaps.

#### E. Character

Character refers to the tenderness of the frozen peas, including snaps.

#### 1. Good Character

The units are tender and are practically uniform in texture and tenderness.

#### 2. Reasonably Good Character

The units are reasonably tender and may be variable in texture and tenderness; and the cotyledons may be mealy or firm but not hard.

# F. Color Defective

A unit that varies markedly from the color that is normally expected for the variety and grade.

#### G. Defect

Any nonconformance with a specified requirement.

#### H. Dissimilar Varieties

In single types only, peas that are markedly different varietal colors and/or shapes. "Dissimilar varieties" is not applicable to snaps in the style of frozen peas with snaps.

# I. Harmless Extraneous Vegetable Material

# 1. In the Style of Frozen Peas

- a. Class 1. Hulls or pieces of unshelled pods, leaves, small tender stems, or other similar vegetable material.
  - b. Class 2. Coarse, fibrous units of vegetable material that are harmless.

# 2. In the Style of Frozen Peas with Snaps

- a. Class 1. Leaves, small tender stems, or other similar vegetable material, except snaps.
  - b. Class 2. Coarse, fibrous units of vegetable material that are harmless.

#### J. Flavor and Odor

#### 1. Good Flavor and Odor

The product, after cooking, has a good, characteristic normal flavor and odor and is free from objectionable flavors and objectionable odors of any kind.

# 2. Reasonably Good Flavor and Odor

The product, after cooking, may be lacking in good flavor but is free from objectionable flavors and objectionable odors of any kind.

#### K. Grit

Sand, silt, or other earthy materials.

## L. Sample

The number of sample units to be used for inspection of a lot.

# M. Sample Unit

The amount of product specified to be used for inspection. It may be

1. The entire contents of a container

- 2. A portion of the contents of a container
- 3. A combination of the contents of two or more containers
- 4. A portion of unpacked product

#### N. Shriveled

A unit that is seriously wrinkled in appearance, including snaps.

# O. Snap

A succulent, unshelled pod of the field pea or black-eyed pea plant or small sieve round type succulent pods of the green bean plant that should be able to pass through the openings of a No. 3 sieve.

#### P. Unit

Any individual frozen pea, or any individual succulent, unshelled pod.

#### VII. INSPECTION CONSIDERATIONS WHEN USING THE DEFINITIONS

The USDA has provided some explanation for the above terms during the inspection of a processing establishment.

# A. Overall Appearance

Judge the prerequisite quality factor of overall appearance on the basis of brightness and dullness. Uniformity of color is not required. Evaluate the color of snaps, in the style of frozen peas with snaps, under the prerequisite factor of overall appearance. Consider off-color snaps as to their effect on the overall appearance of the sample unit. Snaps should be green and succulent pods. Consider any snaps that possess colors that indicate advanced maturity of pods under the factor overall appearance.

#### B. Blemished

Green units of field shelled peas (mechanically harvested) often oxidize and turn brown if held too long before processing. When the units are noticeably discolored, classify as blemished.

Cowpea curculio damage to field peas may occur as visible holes eaten into the cotyledons or discoloration, commonly called weevil sting. Damage is either insignificant or a defect that is counted. It depends on the extent to which the damage is noticeable. Generally, classify units affected by larva holes or dark-colored stings as blemished. Slight discoloration is insignificant. Sometimes Crowder peas develop an objectionable condition during periods of excessive rainfall at harvest. The peas take on an extreme rusty-brown color. Classify this objectionable discoloration as blemished.

#### C. Broken

Mechanical harvesting increases loose skins and broken cotyledons. Some varietal types of field peas, especially cream peas, are more subject to mechanical damage than other varietal types.

Sprouted peas often occur in the sample unit. If the pea is damaged, noticeably, by sprouting, it and the sprout are classified as broken. Include detached sprouts (loose sprouts) with other broken material in the sample unit and weigh.

Determine broken peas on a weight basis. After making several weighings of broken peas, use estimation to judge the amount of broken peas in the sample unit. If the sample unit is borderline, actual weight is advised.

#### D. General Character

General character is a prerequisite quality factor. Use it as a "stopper" if a sample unit meets all other quality factors but is obviously processed from peas that are too mature for good quality. Character is not necessarily related to the number of color attributes. Some "green" peas are hard after cooking 40 minutes. Other sample units with few "green" peas are tender.

Peas. Mechanically harvested field peas normally contain some "seed-dry" peas. Allow for occasional seed-dry peas to avoid being overly critical. In "good character" any seed-dry peas should blend well with the overall palatability of the cooked sample unit. When excessive seed-dry peas are present in the sample unit, its character is grade B, or substandard, depending on the quantity and tenderness of the firm and hard peas.

Snaps. Immature, succulent pods are required as the garnish for frozen peas with snaps. Character is applicable to snaps. However, snaps do not have the same tenderness as pods of other legume plants, such as green beans. Make allowances for the natural characteristics of the field pea pods. Cooking procedure. It is not intended that each sample unit need be cooked for determination of character. Individual judgment should determine the number of sample units to cook. However, cook enough sample units to get a good cross section of character.

## E. Harmless Extraneous Vegetable Material (HEVM)

General. Mechanically harvested peas contain large amounts of HEVM, principally pod and stem material. Shakers, air blasts, and water flotation equipment are used to remove most of this material. Hand-picking on the sorting belt is used for final HEVM cleanup. Without hand-picking, or sorting, the product will rarely make grade.

*Insignificant HEVM*. Consider small, tender, units of the placental part of the pod (connects the pea to the pod) insignificant.

HEVM that is counted. Each individual piece is one defect. Do not reassemble pieces to approximate one piece of pod or pod material.

Unstemmed snaps. In frozen peas with snaps, count each piece of unstemmed snap material as one class 1 HEVM defect. In frozen peas, count each unstemmed snap only once. The stem and pod are related defects and are not counted as two separate defects.

Frozen peas with snaps. If the sample fails the criteria for the style of frozen peas with snaps, don't recount pieces of pod material as HEVM. Consider the sample as failing the requirements for style only.

Hard, woody material. Count hard, woody material as harmful. Beware of handling objectional weed material that is not HEVM, such as foxtail seed heads.

Large units of HEVM. If an otherwise class 1 piece of HEVM is extremely objectionable because of its large size, count the unit as class 2 HEVM.

Other succulent vegetable material. Count other succulent vegetable material that detracts from the overall appearance of the sample unit, such as squash, carrots, or corn, as class 1 HEVM. In the absence of other class 1 HEVM, more of the alien vegetables are permitted. In the presence of other class 1 HEVM, less of the alien vegetables are permitted.

#### F. Shriveled

Field shelled peas lose moisture rapidly. The peas shrink in size. Once the peas are cleaned and placed in holding tanks, filled with water, they absorb moisture and swell to their original size. Don't count peas with slightly wrinkled skin as shriveled.

### G. Snaps

Consider two or more parts of a split pod as one snap in counting snaps for determination of style. Reassemble the pods to their approximate original shape, or the shape of the predominant sized snap in the sample unit. Don't use this procedure for HEVM.

#### VIII. 7 CFR 52.1665. SAMPLE UNIT SIZE

Compliance with requirements for all factors of quality is based on the following sample unit sizes:

- 1. White Acre—5 ounces (141.75 grams)
- 2. All other types—10 ounces (283.5 grams)

#### IX. 7 CFR 52.1666. GRADES

U.S. Grade A is the quality of frozen peas that meets the following prerequisites:

- 1. Has a good appearance
- 2. Has a good flavor and odor
- 3. Is practically free from grit
- 4. Has a good character
- 5. Weight of broken peas does not exceed 0.25 ounce (7.1 grams) for "White Acre" peas and does not exceed 0.5 ounce (14.2 grams) for all other types.

and is within the limits for defects as classified in Table 1 and specified in Table 2.

U.S. Grade B is the quality of frozen peas that meets the following prerequisites

1. Has a reasonably good appearance

- 2. Has a reasonably good flavor and odor
- 3. Is practically free from grit
- 4. Has a reasonably good character
- 5. Weight of broken peas does not exceed 0.5 ounce (14.2 grams) for "White Acre" peas and 1 ounce (28.35 grams) for all other types

and is within the limits for defects as classified in Table 1 and specified in Table 2
Substandard is the quality of frozen peas that fails to meet the requirements for U.S.
Grade B.

#### X. 7 CFR 52.1667. FACTORS OF QUALITY

The grade of a sample of frozen peas is based on compliance with the prerequisites specified in 7 CFR 52.1666 and with limits for the following quality factors:

- 1. Dissimilar varieties and shriveled units
- 2. Harmless extraneous vegetable material
- 3. Blemished units
- 4. Color defectives

#### XI. 7 CFR 52.1668. CLASSIFICATION OF DEFECTS

See Tables 1 and 2.

#### XII. 7 CFR 52.1669. SAMPLE SIZE

The sample size used to determine whether the requirements of these standards are met shall be as specified in the sampling plans and procedures in the Regulations Governing Inspection and Certification of Processed Fruits and Vegetables, Processed Products Thereof, and Certain Other Processed Products (7 CFR 52.1 through 52.83).

# XIII. 7 CFR 52.1670. ACCEPTANCE CRITERIA

# A. Quality Factors

A lot of frozen field peas and black-eyed peas is considered as meeting the requirements for quality if

- 1. The prerequisites specified in 7 CFR 52.1666 are met.
- 2. The Acceptance Numbers in Table 1 or 2 in 7 CFR 52.1667, as applicable, are not exceeded.

**Table 1** AQL's and Tolerances (Tol.) for Defects in Frozen Peas (Except "White Acre") Based on 700 Units of Product for 13 Sample Units,  $700 \times 13 = 9100$  Units

Sample units × sample unit size		$1 \times 700$	$3 \times 700$	6 × 700	$13 \times 700$	$21 \times 700$	$29 \times 700$	
Units of product			700	2100	4200	9100	14700	20300
Defects	AQL	TOL						
	GRAI	DE A			ACCEPTA	ANCE NUMBERS		
Blemished	4.3	4.6	39	106	202	424	674	922
EVM (minor)	0.575	0.7	7	18	32	64	99	134
EVM (major)	0.218	0.3	3	8	14	27	41	55
Dissimilar varieties & shriveled units	3.7	4.0	34	92	176	367	582	796
Color defective <sup>a</sup>	9.9	10.4	83	231	450	950	1,518	2,083
Color defective <sup>b</sup>	16.4	17.0	131	372	728	1,550	2,484	3,416
GRADE B					ACCEPTA	ANCE NUMBERS		
Blemished	6.6	7.0	57	158	304	641	1,022	1,400
EVM (minor)	1.12	1.3	12	31	58	118	186	252
EVM (major)	0.486	0.6	6	15	28	55	85	115
Dissimilar varieties & shriveled units	5.0	5.4	45	122	234	490	780	1.068

<sup>&</sup>lt;sup>a</sup>For black-eyed peas, cream peas, field peas, and mixed types only.

<sup>&</sup>lt;sup>b</sup>For Crowder Peas only.

**Table 2** AQL's and Tolerances (Tol.) for Defects in "White Acre" Frozen Peas Based on 1400 Units of Product for 13 Sample Units,  $1400 \times 13 = 18200$  Units

Sample units × sample unit size  Units of product			$1 \times 1400$	3 × 1400	$6 \times 1400$	$13 \times 1400$	$21 \times 1400$	29 × 1400
			1400	4200	8400	18200	29400	40600
Defects	AQL	TOL						
	GRADE A				ACCEPTA	NCE NUMBERS		
Blemished	2.13	2.3	39	105	201	4200	667	913
EVM (minor)	0.297	0.36	7	18	33	66	102	138
EVM (major)	0.1	0.14	3	7	13	25	38	51
Dissimilar varieties & shriveled units	1.84	2.0	34	92	175	365	579	792
Color defective	4.9	5.2	82	229	445	941	1,503	2,063
	GRADE B				ACCEPTA	NCE NUMBERS		
Blemished	3.3	3.5	57	158	304	641	1,022	1,400
EVM (minor)	0.548	0.64	12	31	57	116	182	247
EVM (major)	0.233	0.29	6	15	27	53	82	110
Dissimilar varieties & shriveled units	2.5	2.7	45	122	234	490	780	1,068

# B. Single Sample Unit

Each unofficial sample unit submitted for quality evaluation will be treated individually and is considered as meeting requirements for quality and style if

- 1. The prerequisites specified in 7 CFR 52.1666 are met.
- 2. The Acceptable Quality Levels (AQL's) in Tables 1 and 2 in 7 CFR 52.1667, as applicable, are not exceeded.

# **27**

# Frozen Fruits and Fruit Juices: Product Description

# **Peggy Stanfield**

Dietetic Resources, Twin Falls, Idaho, U.S.A.

#### I. FROZEN FRUITS

# A. Apples

Frozen apples are prepared from sound, properly ripened fruit of *Malus sylvestris* (*Pyrus malus*); are peeled, cored, trimmed, sliced, sorted, and washed; are properly drained before filling into containers; may be packed with or without the addition of a nutritive sweetening ingredient and any other legally permissible ingredients; and are frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation of the product.

Styles of frozen apples: Slices means frozen apples consisting of slices of apples cut longitudinally and radially from the core axis.

# B. Apricots

Ingredients: Apricots are the food prepared from mature apricots of one of the optional styles specified, which may be packed as solid pack or in one of the optional packing media specified. Such food may also contain one or any combination of two or more of the following safe and suitable optional ingredients:

- 1. Natural and artificial flavors
- 2. Spice
- 3. Vinegar, lemon juice, or organic acids
- 4. Apricot pits, except in the case of unpeeled whole apricots and peeled whole apricots, in a quantity not more than 1 apricot pit to each 227 grams (8 ounces) of finished frozen apricots
- 5. Apricot kernels, except in the case of unpeeled whole apricots and peeled whole apricots, and except when an optional ingredient is used
- 6. Ascorbic acid in an amount no greater than necessary to preserve color

Such food is sealed in a container and before or after sealing is so processed by heat as to prevent spoilage.

# 1. Optional Styles of the Apricot Ingredients

The optional styles of the apricot ingredients are peeled or unpeeled: (a) whole, (b) halves, (c) quarters, (d) slices, (e) pieces or irregular pieces.

Each such ingredient, except in the cases of unpeeled whole apricots and peeled whole apricots, is pitted.

#### 2. Packing Media

The optional packing media are (a) water, (b) fruit juice(s) and water, and (c) fruit juice(s).

Such packing media may be used as such, or any one or any combination of two or more safe and suitable nutritive carbohydrate sweetener(s) may be added. When a sweetener is added as a part of any such liquid packing medium, the density range of the resulting packing medium expressed as percent by weight of sucrose (°Brix) should be designated by the appropriate name for the respective density ranges, namely:

When the density of the solution is 10% or more but less than 16%, the medium should be designated as "slightly sweetened water" or "extra light sirup," "slightly sweetened fruit juice(s) and water" or "slightly sweetened fruit juice(s)," as the case may be.

When the density of the solution is 16% or more but less than 21%, the medium should be designated as "light sirup," "lightly sweetened fruit juice(s) and water," or "lightly sweetened fruit juice(s)," as the case may be. When the density of the solution is 21% or more but less than 25%, the medium should be designated as "heavy sirup," "heavily sweetened fruit juice(s) and water," or "heavily sweetened fruit juice(s)," as the case may be.

When the density of the solution is 25% or more but not more than 40%, the medium should be designated as "extra heavy sirup," "extra heavily sweetened fruit juice(s) and water," or "extra heavily sweetened fruit juice(s)," as the case may be.

# 3. Labeling Requirements

The name of the food is apricots. The name of the food should also include a declaration of any flavoring that characterizes the product and a declaration of any spice or seasoning that characterizes the product; for example, "Spice Added," or in lieu of the word "Spice," the common name of the spice, e.g., "Seasoned with Vinegar" or "Seasoned with Apricot Kernels."

When two or more of the optional ingredients specified are used, such words may be combined, as for example, "Seasoned with Cider Vinegar, Cloves, Cinnamon Oil, and Apricot Kernels."

The style of the apricot ingredient and the name of the packing medium preceded by "In" or "Packed in" or the words "solid pack," where applicable, should be included as part of the name or in close proximity to the name of the food, except that pieces or irregular pieces should be designated "Pieces," "Irregular pieces," or "Mixed pieces of irregular sizes and shapes."

The style of the apricot ingredient should be preceded or followed by "Unpeeled" or "Peeled," as the case may be. "Halves" may be alternatively designated "Halved," "Quarters" as "Quartered," and "Slices" as "Sliced." When the packing medium is prepared with a sweetener(s) that imparts a taste, flavor, or other characteristic to the finished food in addition to sweetness, the name of the packing medium should be accompanied by the name of such sweetener(s), for example, in the case of a mixture of

brown sugar and honey, an appropriate statement would be "—— sirup of brown sugar and honey" the blank to be filled in with the word "light," "heavy," or "extra heavy," as the case may be.

When the liquid portion of the packing media consists of fruit juice(s), such juice(s) should be designated in the name of the packing medium as:

In the case of a single fruit juice, the name of the juice should be used in lieu of the word "fruit."

In the case of a combination of two or more fruit juices, the names of the juices in the order of predominance by weight should be used in lieu of the word "fruit" in the name of the packing medium.

In the case of a single fruit juice or a combination of two or more fruit juices any of which are made from concentrate(s), the words "from concentrate(s)" should follow the word "juice(s)" in the name of the packing medium and in the name(s) of such juice(s) when declared as specified.

Whenever the names of the fruit juices used do not appear in the name of the packing medium, such names and the words "from concentrate," should appear in an ingredient statement.

#### 4. Label Declaration

Each of the ingredients used in the food should be declared on the label.

Frozen apricots are prepared from sound, mature, fresh, peeled or unpeeled fruit of any commercial variety of apricot, which are sorted, washed, and may be trimmed to assure a clean and wholesome product. The apricots are properly drained of excess water before filling into containers; may be packed with the addition of nutritive sweetening ingredient(s) (including sirup and/or sirup containing pureed apricots) and/or suitable antioxidant ingredient(s) and/or any other legally permissible ingredients(s).

The apricots are prepared and frozen in accordance with good commercial practice and are maintained at temperatures necessary for the preservation of the product.

#### 5. Styles of Frozen Apricots

*Halves* are cut approximately in half along the suture from stem to apex and the pit is removed.

Quarters are apricot halves cut into two approximately equal parts.

Slices are apricot halves cut into sectors smaller than quarters.

Diced are apricots cut into approximate cubes.

Cuts are apricots that are cut in such a manner as to change the original conformation and do not meet any of the foregoing styles.

Machine-pitted means mechanically pitted in such a manner as to substantially destroy the conformation of the fruit in removing the pit.

#### C. Berries

Frozen berries are prepared from the properly ripened fresh fruit of the plant (genus *Rubus*); are stemmed and cleaned, may be packed with or without packing media, and are frozen and stored at temperatures necessary for the preservation of the product.

# 1. Types of Frozen Berries

Blackberries

Boysenberries

**Dewberries** 

Loganberries

Youngberries

Other similar types, such as nectar berries

#### 2. Blueberries

a. Product description. Frozen blueberries are prepared from sound, properly ripened fresh fruit of the blueberry bush (genus Vaccinium), including species or varieties often called huckleberries, but not of the genus Gaylussacia; they are cleaned and stemmed, are properly washed, are packed with or without packing media, and are frozen and maintained at temperatures necessary for the preservation of the product.

Types of frozen blueberries: (a) native or wild type; (b) cultivated type.

#### D. Red Tart Pitted Cherries

Frozen red tart pitted cherries are the foods prepared from properly matured cherries of the domestic (*Prunus cerasus*) red sour varietal group that have been washed, pitted, sorted, and properly drained; they may be packed with or without a nutritive sweetened packing medium or any other substance permitted under federal regulations and are frozen and stored at temperatures necessary for the preservation of the product.

#### II. FROZEN JUICES

# A. Apple Juice

Frozen concentrated apple juice is prepared from the unfermented, unsweetened, unacidified liquid obtained from the first pressing of properly prepared, sound, clean, mature, fresh apples, and/or parts thereof by good commercial processes. The juice is clarified and concentrated to at least 22.9°Brix. The apple juice concentrate so prepared, with or without the addition of legal ingredients, is packed and frozen in accordance with good commercial practice and maintained at temperatures necessary for the preservation of the product.

Brix requirements. Brix value of the finished concentrate should not be less than the following for the respective dilution factor of frozen concentrated apple juice:

Dilution Factor Value of Concentrate: minimum Brix (degrees)

- 1 plus 1 22.9
- 2 plus 1 33.0
- 3 plus 1 42.2
- 4 plus 1 50.8
- 5 plus 1 58.8
- 6 plus 1 66.3
- 7 plus 1 73.3

# B. Lemon Juice (for Preparing Frozen Concentrate for Lemonade)

Lemon juice is the unfermented juice, obtained by mechanical process, from sound, mature lemons [Citrus limon (L.) Burm. f.), from which seeds (except embryonic seeds and small fragments of seed which cannot be separated by good manufacturing practice) and excess pulp are removed. The juice may be adjusted by the addition of the optional concentrated lemon juice ingredient in such quantity that the increase in acidity, calculated as anhydrous citric acid, does not exceed 15% of the acidity of the finished food. The lemon oil and lemon essence (derived from lemons) content may be adjusted in accordance with good manufacturing practice. The juice may have been concentrated and later reconstituted. When prepared from concentrated lemon juice, the finished food contains not less than 6%, by weight, of soluble solids taken as the refractometric sucrose value (of the filtrate), corrected to 20°C, but uncorrected for acidity and has a titratable acidity content of not less than 4.5%, by weight, calculated as anhydrous citrus acid.

The food may contain one or any combination of the safe and suitable optional ingredients. Lemon juice may be preserved by heat sterilization (canning), refrigeration, freezing, or by the addition of safe and suitable preservatives. When sealed in a container to be held at ambient temperatures, it is preserved by the addition of safe and suitable preservatives or so processed by heat, before or after sealing, as to prevent spoilage.

# 1. Optional Ingredients

The optional safe and suitable ingredients are (a) concentrated lemon juice (lemon juice from which part of the water has been removed), (b) water and/or lemon juice to reconstitute concentrated lemon juice in the manufacture of lemon juice from concentrate, and (c) preservatives.

# 2. Labeling

The name of the food is

- "Lemon juice" if the food is prepared from unconcentrated, undiluted liquid extracted from mature lemons; or if the food is prepared from unconcentrated, undiluted liquid extracted from mature lemons to which concentrated lemon juice is added to adjust acidity.
- "Lemon juice from concentrate" or "reconstituted lemon juice" if the food is prepared from concentrated lemon juice and water and/or lemon juice; or if the food is prepared from lemon juice from concentrate and lemon juice.

Frozen concentrate for lemonade is the frozen food prepared from one or both of the lemon juice ingredients together with one or any mixture of safe and suitable nutritive carbohydrate sweeteners. The product contains not less than 48.0% by weight of soluble solids taken as the sucrose value.

When the product is diluted according to directions for making lemonade which should appear on the label, the acidity of the lemonade, calculated as anhydrous citric acid, should be not less than 0.70 gram per 100 milliliters, and the soluble solids should be not less than 10.5% by weight.

#### 3. The Lemon Juice Ingredients

Lemon juice or frozen lemon juice or a mixture of these.

Concentrated lemon juice or frozen concentrated lemon juice or a mixture of these. For this purpose, lemon juice is the undiluted juice expressed from mature lemons of an acid variety, and concentrated lemon juice is lemon juice from which part of the water has been removed. In the preparation of the lemon juice ingredients, the lemon oil content may be adjusted by the addition of lemon oil or concentrated lemon oil in accordance with good manufacturing practice, and the lemon pulp in the juice as expressed may be left in the juice or may be separated.

Lemon pulp that has been separated, which may have been preserved by freezing, may be added in preparing frozen concentrate for lemonade, provided that the amount of pulp added does not raise the proportion of pulp in the finished food to a level in excess of that which would be present by using lemon juice ingredients from which pulp has not been separated. The lemon juice ingredients may be treated by heat, either before or after the other ingredients are added, to reduce the enzymatic activity and the number of viable microorganisms.

# C. Frozen Concentrate for Artificially Sweetened Lemonade

Frozen concentrate for artificially sweetened lemonade conforms to the description for frozen concentrate for lemonade, except that in lieu of nutritive sweeteners it is sweetened with one or more of the artificial sweetening ingredients permitted by law, and the soluble solids specifications do not apply. When the product is diluted according to directions that should appear on the label, the acidity of the artificially sweetened lemonade, calculated as anhydrous citric acid, should be not less than 0.70 gram per 100 milliliters. It may contain one or more safe and suitable dispersing ingredients serving the function of distributing the lemon oil throughout the food. It may also contain one or more safe and suitable thickening ingredients. Such dispersing and thickening ingredients are not legal food additives.

The name of the food is "Frozen concentrate for artificially sweetened lemonade." The words "artificially sweetened" should be of the same size and style of type as the word "lemonade." If an optional thickening or dispersing ingredient is used, the label should bear the statement "———— added" or "with added ————," the blank being filled in with the common name of the thickening or dispersing agent used. Such statement should be set forth on the label with such prominence and conspicuousness as to render it likely to be read and understood by the ordinary individual under customary conditions of purchase.

#### D. Frozen Concentrate for Colored Lemonade

Frozen concentrate for colored lemonade conforms to the description for frozen concentrate for lemonade, except that it is colored with a safe and suitable fruit juice, vegetable juice, or any such juice in concentrated form, or with any other legal color additive ingredient suitable for use in food, including legal artificial coloring.

#### E. Grapefruit Juice

Grapefruit juice is the unfermented juice, intended for direct consumption, obtained by mechanical process from sound, mature grapefruit (Citrus paradisi Macfadyen) from

which seeds and peel (except embryonic seeds and small fragments of seeds and peel that cannot be separated by good manufacturing practice) and excess pulp are removed and to which may be added not more than 10% by volume of the unfermented juice obtained from mature hybrids of grapefruit. The juice may be adjusted by the addition of the optional concentrated grapefruit juice ingredients specified, but the quantity of such concentrated grapefruit juice ingredient added should not contribute more than 15% of the grapefruit juice soluble solids in the finished food. The grapefruit pulp, grapefruit oil, and grapefuit essence (components derived from grapefruit) content may be adjusted in accordance with good manufacturing practice. The juice may have been concentrated and later reconstituted with water suitable for the purpose of maintaining essential composition and quality factors of the juice. It may be sweetened with the dry nutritive sweeteners. If the grapefruit juice is prepared from concentrate, such sweeteners in liquid form also may be used. When prepared from concentrated grapefruit juice, exclusive of added sweeteners, the finished food contains not less than 10%, by weight, of soluble solids taken as the refractometric sucrose value (of the filtrate), corrected to 20°C, and corrected for acidity by adding  $(0.012 + 0.193x - 0.0004x \ge 1)$ , where x equals the percent anhydrous citric acid in the sample, to the refractometrically obtained sucrose value. Grapefruit juice, as defined in this paragraph, may be preserved by heat sterlization (canning), refrigeration, or freezing. When sealed in a container to be held at ambient temperatures, it is so processed by heat, before or after sealing, as to prevent spoilage.

# 1. Optional Ingredients

The optional ingredients are (a) concentrated grapefruit juice (grapefruit juice from which part of the water has been removed); (b) water and/or grapefruit juice to reconstitute concentrated grapefruit juice in the manufacture of grapefruit juice from concentrate; (c) one or any combination of two or more of the dry or liquid forms of sugar, invert sugar sirup, dextrose, glucose sirup, and fructose.

Labeling. The name of the food is

"Grapefruit juice" if the food is prepared from unconcentrated, undiluted liquid extracted from mature grapefruit, or if the food is prepared from unconcentrated, undiluted liquid extracted from mature grapefruit to which concentrated grapefruit juice is added to adjust soluble solids.

"Grapefruit juice from concentrate" if the food is prepared from concentrated grapefruit juice and water and/or grapefruit juice; or if the food is prepared from grapefuit juice from concentrate and grapefruit juice. The words "from concentrate" should be shown in letters not less than one-half the height of the letters in the words "Grapefruit juice."

If any nutritive sweetener is added, the principal display panel of the label should bear the statement "Sweetener added." If no sweetener is added, the word "Unsweetened" may immediately precede or follow the words "Grapefruit Juice" or "Grapefruit Juice from Concentrate."

# F. Orange Juice

Orange juice is the unfermented juice obtained from mature oranges of the species *Citrus sinensis* or of the citrus hybrid commonly called "Ambersweet"  $[\frac{1}{2}]$  *Citrus sinensis* ×  $\frac{3}{8}$  *Citrus reticulata* ×  $\frac{1}{8}$  *Citrus paradisi* (USDA Selection: 1-100-29: 1972 Whitmore

Foundation Farm)]. Seeds (except embryonic seeds and small fragments of seeds that cannot be separated by current good manufacturing practice) and excess pulp are removed. The juice may be chilled, but it is not frozen.

The name of the food is "orange juice." The name "orange juice" may be preceded on the label by the varietal name of the oranges used, and if the oranges grew in a single State, the name of such State may be included in the name, as for example, "California Valencia orange juice."

### G. Pasteurized Orange Juice

Pasteurized orange juice is the food prepared from unfermented juice obtained from mature oranges, to which may be added not more than 10% by volume of the unfermented juice obtained from mature oranges of the species Citrus reticulata or Citrus reticulata hybrids. Seeds (except embryonic seeds and small fragments of seeds that cannot be separated by good manufacturing practice) are removed, and pulp and orange oil may be adjusted in accordance with good manufacturing practice. If the adjustment involves the addition of pulp, then such pulp should not be of the washed or spent type. The solids may be adjusted by the addition of one or more of the optional concentrated orange juice ingredients. One or more of the optional sweetening ingredients may be added in a quantity reasonably necessary to raise the Brix or the Brix-acid ratio to any point within the normal range usually found in unfermented juice obtained from mature oranges. The orange juice is so treated by heat as to reduce substantially the enzymatic activity and the number of viable microorganisms. Either before or after such heat treatment, all or a part of the product may be frozen. The finished pasteurized orange juice contains not less than 10.5% by weight of orange juice soluble solids, exclusive of the solids of any added optional sweetening ingredients, and the ratio of the Brix hydrometer reading to the grams of anhydrous citric acid per 100 milliliters of juice is not less than 10 to 1.

The optional concentrated orange juice ingredients are frozen concentrated orange juice and concentrated orange juice for manufacturing when made from mature oranges; but the quantity of such concentrated orange juice ingredients added should not contribute more than one-fourth of the total orange juice solids in the finished pasteurized orange juice.

The optional sweetening ingredients referred to are sugar, invert sugar, dextrose, dried corn sirup, dried glucose sirup.

The name of the food is "Pasteurized orange juice." If the food is filled into containers and preserved by freezing, the label should bear the name "Frozen pasteurized orange juice." The words "pasteurized" or "frozen pasteurized" should be shown on labels in letters not less than one-half the height of the letters in the words "orange juice."

If the pasteurized orange juice is filled into containers and refrigerated, the label should bear the name of the food, "chilled pasteurized orange juice." If it does not purport to be either canned orange juice or frozen pasteurized orange juice, the word "chilled" may be omitted from the name. The words "pasteurized" or "chilled pasteurized" should be shown in letters not less than one-half the height of the letters in the words "orange juice."

# H. Frozen Concentrated Orange Juice

Frozen concentrated orange juice is the food prepared by removing water from the juice of mature oranges, to which may be added unfermented juice obtained from mature oranges

of the species Citrus reticulata, other Citrus reticulata hybrids, or of Citrus aurantium, or both. However, in the unconcentrated blend, the volume of juice from Citrus reticulata or Citrus reticulata hybrids should not exceed 10%, and from Citrus aurantium should not exceed 5%. The concentrate so obtained is frozen. In its preparation, seeds (except embryonic seeds and small fragments of seeds that cannot be separated by good manufacturing practice) and excess pulp are removed, and a properly prepared water extract of the excess pulp so removed may be added. Orange oil, orange pulp, orange essence (obtained from orange juice), orange juice and other orange juice concentrate or concentrated orange juice for manufacturing (when made from mature oranges), water, and one or more of the optional sweetening ingredients may be added to adjust the final composition. The juice of Citrus reticulata and Citrus aurantium, as permitted by this paragraph, may be added in single strength or concentrated form prior to concentration of the Citrus sinensis juice, or in concentrated form during adjustment of the composition of the finished food. The addition of concentrated juice from Citrus reticulata or Citrus aurantium, or both, should not exceed, on a single-strength basis, the 10% maximum for Citrus reticulata and the 5% maximum for Citrus aurantium prescribed by this paragraph. Any of the ingredients of the finished concentrate may have been so treated by heat as to reduce substantially the enzymatic activity and the number of viable microorganisms. The finished food is of such concentration that when diluted according to label directions the diluted article will contain not less than 11.8% by weight of orange juice soluble solids, exclusive of the solids of any added optional sweetening ingredients. The dilution ratio should be not less than 3 plus 1. The term "dilution ratio" means the whole number of volumes of water per volume of frozen concentrate required to produce orange juice from concentrate having orange juice soluble solids of not less than 11.8% by weight exclusive of the solids of any added optional sweetening ingredients.

The optional sweetening ingredients are sugar, sugar sirup, invert sugar, invert sugar sirup, dextrose, corn sirup, dried corn sirup, glucose sirup, and dried glucose sirup.

If one or more of the sweetening ingredients are added to the frozen concentrated orange juice, the label should bear the statement "—— added," the blank being filled in with the name or an appropriate combination of names of the sweetening ingredients used. However, the name "sweetener" may be used in lieu of the specific name or names of the sweetening ingredients.

## I. Reduced Acid Frozen Concentrated Orange Juice

Reduced-acid frozen concentrated orange juice is the food that complies with the requirements for composition and label declaration of ingredients prescribed for frozen

concentrated orange juice except that it may not contain any added sweetening ingredient. A process involving the legal use of anionic ion-exchange resins is used to reduce the acidity of the food so that the ratio of the Brix reading to the grams of acid, expressed as anhydrous citric acid, per 100 grams of juice is not less than 21 to 1 or more than 26 to 1.

The name of the food is "Reduced acid frozen concentrated orange juice."

# J. Orange Juice for Manufacturing

Orange juice for manufacturing is the food prepared for further manufacturing use. It is prepared from unfermented juice obtained from oranges as provided earlier, except that the oranges may deviate from the standards for maturity in that they are below the minimum for Brix and Brix—acid ratio for such oranges, and to which juice may be added not more than 10% by volume of the unfermented juice obtained from oranges of the species *Citrus reticulata* or *Citrus reticulata* hybrids (except that this limitation should not apply to the hybrid species). Seeds (except embryonic seeds and small fragments of seeds that cannot be separated by good manufacturing practice) are removed, and pulp and orange oil may be adjusted in accordance with good manufacturing practice. If pulp is added it should be other than washed or spent pulp. The juice or portions thereof may be so treated by heat as to reduce substantially the enzymatic activity and number of viable microorganisms, and it may be chilled or frozen, or it may be so treated by heat, either before or after sealing in containers, as to prevent spoilage.

The name of the food is "Orange juice for manufacturing."

# K. Orange Juice with Preservative

Orange juice with preservative is the food prepared for further manufacturing use. It complies with the requirements for composition of orange juice for manufacturing as specified, except that a preservative is added to inhibit spoilage. It may be heat-treated to reduce substantially the enzymatic activity and the number of viable microorganisms.

The preservatives referred to are any safe and suitable preservatives or combinations thereof.

The name of the food is "Orange juice with preservative." Label declaration. Each of the ingredients used in the food should be declared on the label as required by regulations. In addition, the name of each preservative should be proceeded by a statement of the percent by weight of the preservative used. If the food is packed in container sizes that are less than 19 liters (5 gallons), the label should bear a statement indicating that the food is for further manufacturing use only.

Wherever the name of the food appears on the label so conspicuously as to be easily seen under customary conditions of purchase, the statement for naming the preservative ingredient used should immediately and conspicuously precede or follow the name of the food, without intervening written, printed, or graphic matter.

# L. Concentrated Orange Juice for Manufacturing

Concentrated orange juice for manufacturing is the food that complies with the requirements of composition and label declaration of ingredients prescribed, except that it is either not frozen or is less concentrated, or both, and the oranges from which the juice is obtained may deviate from the standards for maturity in that they are below the

minimum Brix and Brix-acid ratio for such oranges: However, the concentration of orange juice soluble solids should not be less than 20 deg. Brix.

The name of the food is "Concentrated orange juice for manufacturing, ——" or "—— orange juice concentrate for manufacturing," the blank being filled in with the figure showing the concentration of orange juice soluble solids in degrees Brix.

# M. Concentrated Orange Juice with Preservative

(a) Concentrated orange juice with preservative complies with the requirements for composition and labeling of optional ingredients prescribed for concentrated orange juice for manufacturing by Sec. 146.153, except that a preservative is added to inhibit spoilage. (b) The preservatives referred to in paragraph (a) of this section are any safe and suitable preservatives or combinations thereof. (c) The name of the food is "Concentrated orange juice with preservative, ——," the blank being filled in with the figure showing the concentration of orange juice soluble solids in degrees Brix. (d) Label declaration. Each of the ingredients used in the food should be declared on the label as required by the applicable sections of parts 101 and 130 of this chapter. In addition, the name of each preservative should be preceded by a statement of the percent by weight of the preservative used. If the food is packed in container sizes that are less than 19 liters (5 gallons), the label should bear a statement indicating that the food is for further manufacturing use only.

# N. Pineapple Juice

Pineapple juice is the juice, intended for direct consumption, obtained by mechanical process from the flesh or parts thereof, with or without core material, of sound, ripe pineapple (*Ananas comosus* L. Merrill). The juice may have been concentrated and later reconstituted with water suitable for the purpose of maintaining essential composition and quality factors of the juice. Pineapple juice may contain finely divided insoluble solids, but it does not contain pieces of shell, seeds, or other coarse or hard substances or excess pulp. It may be sweetened with any safe and suitable dry nutritive carbohydrate sweetener. However, if the pineapple juice is prepared from concentrate, such sweeteners, in liquid form, also may be used. It may contain added vitamin C in a quantity such that the total vitamin C in each 4 fluid ounces of the finished food amounts to not less than 30 milligrams and not more than 60 milligrams. In the processing of pineapple juice, dimethylpolysiloxane may be employed as a defoaming agent in an amount not greater than 10 parts per million by weight of the finished food. Such food is prepared by heat sterilization, refrigeration, or freezing. When sealed in a container to be held at ambient temperatures, it is so processed by heat, before or after sealing, as to prevent spoilage.

The name of the food is "Pineapple juice" if the juice from which it is prepared has not been concentrated and/or diluted with water. The name of the food is "Pineapple juice from concentrate" if the finished juice has been made from specified pineapple juice. If a nutritive sweetener is added, the label should bear the statement "Sweetener added." If no sweetener is added, the word "Unsweetened" may immediately precede or follow the words "Pineapple juice" or "Pineapple juice from concentrate."

Label declaration. Each of the ingredients used in the food should be declared on the label.

# 1. Quality

The standard of quality for pineapple juice is as follows: (a) The soluble solids content of pineapple juice (exclusive of added sugars) without added water should not be less than 10.5 deg. Brix as determined by refractometer at 20°C uncorrected for acidity and read as degrees Brix on International Sucrose Scales. Where the juice has been obtained using concentrated juice with addition of water, the soluble pineapple juice solids content (exclusive of added sugars) should be not less than 12.8 deg. Brix, uncorrected for acidity and read as degrees Brix on the International Sucrose Scales. The acidity is not more than 1.35 grams of anhydrous citric acid per 100 milliliters of the juice. The ratio of the degrees Brix to total acidity is not less than 12. The quantity of finely divided "insoluble solids" is not less than 5% nor more than 30%.

# O. Blended Grape and Orange Juice

#### 1. Product Description

Frozen concentrated blended grapefruit juice and orange juice is the frozen product prepared from a combination of concentrated, unfermented juices obtained from sound, mature grapefruit (*Citrus paradisi*) and from sound, mature fruit of the sweet orange group (*Citrus sinensis*) and Mandarin group (*Citrus reticulata*), except tangerines. The fruit is prepared by sorting and by washing prior to extraction of the juices to assure a clean product. The juices may be blended upon extraction of such juices or after concentration, and fresh orange juice extracted from sorted and washed fruit, as aforesaid, is admixed to the concentrate. It is recommended that the frozen concentrated blended grapefruit juice and orange juice be composed of the equivalent of not less than 50% orange juice in the reconstituted juice; however, in oranges yielding light-colored juice it is further recommended that as much as the equivalent of 75% orange juice in the reconstituted juice be used. The concentrated juice is packed in accordance with good commercial practice and is frozen and maintained at temperatures necessary for the preservation of the product.

# 2. Styles of Frozen Concentrated Blended Grapefruit Juice and Orange Juice

- a. Style I, Without Sweetening Ingredient Added. The Brix value of the finished concentrate should be not less than 40 degrees nor more than 44 degrees.
- b. Style II, With Sweetening Ingredient Added. The finished concentrate, exclusive of added sweetening ingredient, has a Brix value of not less than 38 degrees; and the finished concentrate, including added sweetening ingredient, should have a Brix value of not less than 40 degrees but not more than 48 degrees.

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# Frozen Guava and Papaya Products

Harvey T. Chan, Jr.
HI Food Technology, Hilo, Hawaii, U.S.A.

# I. FROZEN GUAVA PRODUCTS

#### A. Introduction

The common guava, *Psidium guajava* L., is native to tropical America and widely distributed throughout the tropics. The guava is the most widely known and important fruit plant in the Myrtaceae family. Introduced into Hawaii in about 1790, it flourishes in nearly all parts of the islands at elevations below 3000 ft. The plants have become wild and are considered a noxious weed. Commercial production of the fruit is primarily for manufacture of guava puree from the acid-pink varieties. Under favorable growing conditions, the guava plant develops into a small tree often attaining heights of 30 ft or more. The plant is shallow rooted and has a loose but symmetrical canopy, forming branches close to the ground (1). The bisexual or perfect flowers are white in color and from 1 in. to about 1.5 in. in diameter. The stamens are numerous and the pollen plentiful. Cross-pollination is frequently aided by pollen-carrying insects. Self-pollination is possible, and isolated trees often set satisfactory crops of fruit without cross-pollination.

The fruits, which are round, ovate, or pea-shaped, vary from 1 to 4 in. in diameter and from 2 oz to 1 lb in weight. The skin color of the ripe fruit is yellow, and the flesh color may be white, pink, yellow, salmon, or carmine. The fruits range in flavor from quite sweet to sour. Low-acid cultivars such as "Allahabad Safeda" and "Apple Color," with white flesh, have been developed in India. Mild, sweet dessert types with light pink color for consumption as a fresh fruit are South Africa's "Malherbe" and "Fan Retief" (2).

For processing, fruits with high acid and deep purple or red color are sought, in addition to uniform color, excellent flavor, high acidity, thick flesh, and high yields. The "Beaumont" variety and its decendants "Kahua Kula" and "Waiakea" are the dominant processed pink varieties worldwide. This is due to the early commercialization of "Beaumont," which established the standards for excellent flavor, color, and high yields.

# B. Horticultural Aspects

Under natural conditions in Hawaii, the main guava crop usually ripens from June to September. A smaller distinct crop is produced during the winter season. New and novel techniques for crop cycling of guavas have been recently developed by University of Hawaii horticulturists (3). The techniques employ defoliation, pruning, fertilization,

irrigation, and various combinations of these methods. The concept of crop cycling ensures a continuous yearly supply of fruit for the processors. Crop cycling has been practiced in Hawaii for the past 20 years. Other advantages to crop cycling are the shortening of the harvest period, which reduces labor costs, enables more precise scheduling for the fruit processor, enables reduced warehousing of product, and provides a more recently (fresher) processed product availability to client/buyer and increased opportunity for year-round employment of both field and processing plant workers (4).

Because of the high variability in fruit quality from guava trees propagated from seeds, clonal propagation by vegetative propagation is the preferred method. Plants raised from seeds take about 18 to 24 months to bear fruit, while vegetatively propagated guava trees take about 6 months. Bud grafting is the preferred method of asexual propagation because of its high rate of success. Rootstocks are commonly grown from seeds. Seedlings are usually suitable for grafting when they are about 5 to 7 months old and about 1 cm in diameter and 30 cm in height (1).

The guava is a hardy plant that has adapted to a wide range of soil and climatic conditions in many areas of the tropics and subtropics within latitudes of 35°N and 35°S. It is cultivated in areas of Southern California, where it is subject to seasonal frost cycles that completely defoliate the trees. It is also cultivated in the hot and humid Johore region of Malaysia, where it is growing on spent tin tailings. A good rainfall is essential for sustained vegetative growth and the emergence of new shoots, but prolonged heavy rain during flowering and fruiting may cause flower or fruit drop or fruit splitting and increase the incidence of fruit rot. Heavy rain during fruit ripening may even cause a loss of fruit flavor. Guava plants can withstand mean air temperature ranges from 15 to 45°C but have the highest yields at 23 to 28°C (1).

# C. Harvesting and Yield

Yields of guava vary from 26,000 lbs/acre to as high as 45,000 lbs/acre depending on cultural practices, age of plant, and cultivar. Guava plants reach peak yields within 5 to 7 years and continue to bear fruit well past 25 years. In Hawaii, forty-year-old trees that have been well cared for continue to produce crops. Annually, Hawaii exports in excess of 16 million pounds of guava puree. About 2 million pounds of puree are consumed within the State of Hawaii by its 1.2 million citizens and its 6 to 9 million tourists as guava juice is blended with other fruit juices. The most popular blend is passionfruit, orange, and guava, known as POG.

Guava fruits take about 120 to 140 days from fruit set to reach full maturity. However, the fruits should be picked before they are fully ripe. Ripe fruits are extremely susceptible to fruit fly infestation (*Dacus* sp.) and its resultant infections of fungal and yeast decay. Overripe fruits also invite predation by birds, rodents, and snails, so guava fruits are picked while still slightly green and firm. The fruits are hand harvested, placed into containers, and transported via tractors in shallow field bins of 500 lb capacity to the processing plant, wherein they are usually processed within the day of harvest.

# D. Guava Processing

Processing guava into puree or juice is relatively simple. Commonly available fruit processing equipment is used, and little labor is required. The first step involves a thorough washing of the fruit to remove any adhering soil or contamination and a sorting procedure to remove unsound or immature fruit.

#### E. Guava Puree

Guava puree is used in the manufacture of guava nectar and various juice drink blends and in the preparation of guava jam. Ideally it is a bright pink color that will not require the addition of artificial color in the manufacture of the finished products. The washed, sound fruit is first passed through a chopper or slicer to pulverize the fruit, and this material is fed into a pulper. The pulper removes seeds and fibrous pieces of tissue, forcing the remainder of the product through a perforated stainless steel screen. The holes in the screen should be between 0.033 and 0.045 in. The pulping machine should be fed at a constant rate to ensure efficient operation and also ensuring that the pulping does not run dry, which would cause off-flavors. The pureed material coming from the pulper is next passed through a finisher. The finisher is a piece of machinery identical to the pulper except that it is fitted with a finer mesh screen containing holes between 0.020 to 0.010 in. The finisher fitted with 0.020 screen will remove most of the stone cells from the puree. To remove all of the stone cells, a 0.010 screen and/or a centrifugal separator is recommended. Yield data computed on the basis of a 0.033 in. screen for the pulper and a 0.020 in. screen in the finisher showed 12.0% waste as seed and 5.5% waste as stone cells.

Perhaps the best way to preserve the quality of guava puree is by freezing, and the material passing through the finisher can be packaged and frozen with no further treatment. It is not necessary to heat the product to inactivate the enzymes. However, to comply with recent U.S. Food and Drug regulations regarding pasteurized juices, a short and mild heat treatment that effectively reduces by five log orders (5-decimal reduction) the microbial numbers for  $E.\ coli$  is required. Pasteurization can be accomplished by passing the puree through one of several different heat exchanger configurations. Plate, tubular, and scraped-surface heat exchangers are currently being used. The cooled puree can be frozen directly in a number of types of poly-lined cartons up to 40 lbs, but a fiber box with a plastic bag inside is commonly used and is probably the least expensive. Larger containers such as 55 gal. containers require a prechilling through a slush freezer to lower the puree temperature below 35°F before filling the container. The puree is frozen in a blast freezer at approximately -20°F and stored at 0°F.

Transoceanic shipping tanks (AgMark, TM) that are insulated but with no means of refrigeration are currently being used to ship chilled guava puree produced in Hawaii (Kilueau Agronomics, LLC) to the U.S. mainland. The shipping tankers rely on highbulk, 4,500 gallons, and thick urethane insulation to maintain temperature increases of 1°F or less per day while in transit. The shipping containers are filled at 34°F or less and then topped off with frozen blocks of guava puree. Extra insulation is added to the inlet and outlet fixtures, which reduces heat conduction from ambient conditions. The normal transit time from ports in Hawaii to the U.S. mainland is about 5 or 6 days, so typically the puree arrives at temperatures of 40°F or less. Recent studies have shown that microbial numbers, cfu, such as the total counts of yeast, bacteria, and mold, actually decrease with storage time at temperatures less than 40°F.

# F. Guava Juice

Guava juice is a clear or semiclarified product prepared by the removal of solid pulpy material. The juice can be used in the manufacture of clear guava jelly or in various blended drinks. It will have a light amber or slight pink coloration, since most of the pink pigments in the guava remain with the solid material.

A clear juice is prepared from guava puree that has been depectinized enzymatically. About 0.1% by weight of Pectinol 10 M (or an equivalent amount of any pectin-degrading enzyme) is mixed into the puree at room temperature. Heating of the product to about 120°F will greatly accelerated the enzyme action. After about 1h, clear juice is separated from the red pulp by centrifuging or by pressing in a hydraulic juice press. A batch-type or continous-flow centrifuge can be used on the depectinized puree with no further treatment. If a hydraulic press is used, diatomaceous earth must be mixed into the depectinized puree to facilitate the pressing operation. About 0.5% to 1.0% of a coarse grade of filter aid (Celite 545 or equivalent) or cellulose filter floc is mixed into the puree with a power stirrer. The puree is filled into the nylon press cloths or bags, and juice is expressed by applying hydraulic pressure. This press juice usually contains some suspended solids and must be further clarified in a filter press. The clear juice effluent from the filter press should be heated sufficiently to destroy the pectic enzymes. This is best done in a plate or corrugated or spiral tubular heat exchanger at 195°F for 15 seconds. The actual time-temperature relation will depend on the pectic enzymes used and the pH of the juice. Following the inactivation of the enzymes, the juice may be further clarified by passing through a filter press with addition of a suitable pressing aid such as diatomaceous earth. After clarification, the juice may be frozen in a suitable container or canned and heat processed.

#### G. Guava Concentrate

Guava concentrate can be prepared by using a Centritherm (Alpha Laval) centrifugal vacuum evaporator. This type of evaporator has a short residence time and a low evaporation temperature, which minimizes heat-induced flavor losses. The puree is treated with a pectin-degrading enzyme (Pectinol 10 M), 0.1%, or any other cell-wall-degrading enzymes before concentration. This decreases the consistency (thickness), so a higher degree of concentration can be achieved. The enzyme-treated puree is kept at ambient temperature for 1 hour and then concentrated in the vacuum evaporator. Evaporation was conducted at reduced pressures (62-72 mm Hg) and at a vapor temperature of 108 to 113°F. Guava puree has been concentrated 3.5-fold; clear guava juice from which all the pulp has been removed can be concentrated eight fold or higher. Means of stabilizing these concentrates at refrigerated temperatures (35 to 45°F) have been devised making it possible to transport the concentrates overseas at above-freezing temperatures. The method involves the addition of potassium sorbate to a level of 1,000 ppm to a 2.5-fold concentrate, 22.5°Brix. After five months of storage at 45°F, no gross signs of spoilage were present. Flavor and aroma quality were good and did not deteriorate appreciably until the fourth month in storage (5).

#### H. Frozen Guava Nectar Base

Guava nectar base is a combination of puree and sugar in such proportions that it may be diluted with water by the consumer in the same manner that many other fruit juice concentrates and nectars are prepared. For the Hawaiian palate, an optimum dilution of 2.5 to 3 parts water to 1 part nectar base was determined by a taste panel. The formula for the nectar base was 100 lb guava puree at 7°Brix and 48 lb sugar. Citric acid is added to the mixture to adjust the pH to 3.3–3.5. After the mixture has been blended, it should be pumped through a slush freezer. It should then be filled into suitable containers and frozen immediately at 0°F or below.

# **II. FROZEN PAPAYA PRODUCTS**

#### A. Introduction

The papaya (Carica papaya L.) is a tropical plant grown between the latitudes of 32° north and south. The fruit size ranges from less than 1 lb to 20 lb. The papaya is indigenous to southern Mexico and Costa Rica. It was taken by the Spaniards to Manila in the sixteenth century and reached Malacca shortly afterwards. From there it was introduced to India. It was reported in Zanzibar in the eighteenth century and in Uganda in 1874 (6). The introduction of papaya to Hawaii is usually credited to Don Marin, an early settler and horticulturist, who brought the seeds from the Marquesas Islands in the early 1800s.

Since that early period, papayas have become one of Hawaii's major agricultural export crops. About 5 million pounds of cull papayas are processed per year into puree. Other products are papaya seed dressing, minimal processed papaya cubes, and papaya jams. In the United States the majority of the fresh fruit is consumed ripe. However, in many Asian countries, especially Japan, Malaysia, and Thailand, the fruit is consumed as a grated vegetable while still in the green stage.

# **B.** Horticultural Aspects

# 1. Climatological and Soil Requirements

The papaya tree is able to grow on many different soils that have good drainage and a soil pH of 5.0 to 7.0 with optimal pH between 5.5 to 6.5. At present, 90% of the papayas grown in Hawaii are grown in a rocky volcanic soil called an that is composed primarily of porous lava, volcanic ash, weathered rock material, and some organic matter (7).

The papaya has adapted to a wide range of rainfall conditions in Hawaii, ranging from 1.5 to over 2.5 m/year. Papayas are grown in the Puna area of Hawaii, which experiences rainfalls greater than 2.5 m/year owing to the highly porous nature of the volcanic soils. Most of the papayas grown in Hawaii are in areas considered warm, on lands from a few feet to 500 ft above sea level. Papayas grown at higher elevations with lower temperatures usually produce fruit with lower sugar content and poor market quality.

# 2. Propagation

Papaya plants are usually started with seeds from ripe fruit that have been processed and dried. The seeds are processed by fermentation in a bucket of water for a few days. The fermentation facilitates the removal of the gelatinous seed coat. Addition of cell wall degrading enzymes also assists in the removal of the sarcotesta. The sarcotesta is washed off with vigorous scrubbing and the remaining seed is dried. The seeds are planted directly in the field. Twelve or more seeds are placed in a hole. Germination occurs within 10 to 14 days. The papaya seedlings are thinned out 4 to 6 weeks after germination. Only the three strongest seedlings are allowed to remain. The second and final thinning occurs when the papaya flowers are large enough to determine if the tree is hermaphroditic or female. Only a hermaphroditic tree is selected to grow per planting hole. Fruits from hermaphroditic trees are deemed to be superior in fruit quality, having thicker succulent flesh and more attractive pear shaped fruits.

#### 3. Harvesting and Yield

Papayas at the proper maturity for harvesting show a tinge or more of yellow at the apical end of the fruit. These fruits are harvested manually when the trees are short. With older mature trees, whose fruit are beyond the reach of the picker equipped with a rubber plunger on a 6 to 8 ft bamboo pole, mechanical harvesting aids are employed. These mechanical aids are self-propelled tractors upon which the picker platforms are elevated on bucket booms. Each picker has a fruit conveyor running the length of the boom to a bin. Each bin holds about 900 lb of papayas, and the machine capacity is eight bins. In commercial orchards, papaya trees that are harvested manually are cultivated for 3 years, since after 3 years the trees are too tall for harvesting. Those orchards that are mechanically harvested may be harvested for an additional 1 or 2 years owing to the added height advantage of the harvesting machines.

# 4. Postharvest Handling

Papayas are treated to reduce storage decay by immersing in hot water at 120°F for 20 min, then cooled in running water for 20 min (8). Low-temperature storage 15°C delays color change as well as loss of firmness (9). Papaya is sensitive to chilling. Common visual symptoms are skin pitting, uneven ripening, skin discoloration, formation of lumpy tissue, and susceptibility to fungal rot (10). Susceptibility of heat-treated papayas to chilling injury can be decreased by holding the heat-treated fruit after cooling in a ripening room before cold storage (11).

Disinsectation methods for fruit flies in papayas involve the use of vapor heat and irradiation. In the vapor heat treatment , the papayas are first preconditioned to dry heat [40% relative humidity (RH)] at  $110^{\circ}F$  for 6 to 8 h. The papayas are next subjected to moist heat (100% RH) so that the center temperature of the fruit reaches  $117^{\circ}F$  and is held for 4 h. The fruit are then air-cooled. In the irradiation method, papayas are irradiated at a minimum dose of 250 Gy, which sterilizes the fruit-fly eggs and larvae (12).

#### 5. Papaya Processing

a. Biochemical Changes During Processing and Storage. Several chemical and biochemical changes can occur in processed papaya products during processing and storage. These changes can be classified as enzymatic, nonenzymatic, and microbial.

Enzymatic Changes. Enzymatic changes are generally initiated in the manufacture of papaya puree when the fruit undergoes a pulping operation whereby fruit tissues are disrupted, causing the release and mixing of enzymes and substrate. Several deleterious enzymatic reactions that affect the product can then ensue. Off-flavor and off-odor development is due to enzymatic and microbial activity (13). Butyric, hexanoic, and octanoic acids and their methyl esters were found in purees prepared by commercial methods in which the enzymes had not been inactivated by acidification and heat. A pungent sulfury odor has also been known to evolve from papaya puree, especially puree made from green fruit. Benzyl isothiocyanate (BITC) has been identified to be the cause of the pungent odors in papaya. Benzyl isothiocyanate is formed by the enzymatic (thioglucosidase or myrosinase) hydrolysis of of benzylglucosinolate. Gelation of papaya puree due to pectin esterase activity is an important problem facing a processor. Immediatedly after papaya is pulped, a gel is formed unless certain steps are taken to inhibit or inactivate pectin esterase activity. The formation of gels may be prevented by the use of sucrose (14) or the application of heat and acidification (13). Besides gelling, the

acidity of the puree decreases in pH from 5.2 to 4.6. One of the enzymes responsible for this is acid phosphatase, which catalyzes the hydrolysis of the P—O bond of orthophosphoric monoesters producing ROH and H<sub>3</sub>PO<sub>4</sub>. The release of phosphoric acid lowers the pH (15). Another enzymatic problem facing the processor is the action of invertase, which hydrolyzes sucrose to glucose and fructose. The conversion is rapid in papaya puree, with 50% of the sucrose being hydrolyzed within 2.6 min after the tissue is macerated (16, 17). The conversion of nonreducing sugars to reducing sugars increases the potential susceptibility of processed papaya products to nonenzymatic browning during high-temperature or prolonged storage conditions.

Microbial Changes. Several microbial changes can occur when papaya products are improperly handled or stored. The development of off-flavors and odors is partly due to the emanation of volatile and nonvolatile short chain fatty acids and their methyl esters (13). The presence of sulfury off-odors in papaya products can be attributed to the production of  $H_2S$  and benzylamine owing to the microbial activity of Enterobactor cloacae (18).

Nonenzymatic Changes. The quality and nutritive value of papaya products may also be altered by enzymatic changes occurring during processing (19). Ascorbic acid losses were significant during puree processing (5.5%) and during evaporative concentration (14.3%). Heat applied during the concentration process was responsible for the ascorbic acid loss. The total ascorbic loss from crushed fruit to concentrate was 20.3%.

Absorption spectra differed for total carotenoid extracts of fresh papayas, puree, and concentrated puree. Absorption maximum for the total carotenoid of fresh papaya was at 445 nm, with minor peaks at 469 and 425 nm. After the acidification to pH 3.5 in the processing of puree, the spectrum shifted with increased absorption at 425 nm and decreased absorption at 445 nm. The difference became pronounced after concentration when absorption at 425 nm was clearly the major peak. The hypsochromic effect increased progressively with the processing sequence.

Of the total carotenoids, about 15% cryptoxanthinmonoepoxide was detected in fresh papaya puree, 9.8% in processed puree, and none in papaya concentrate. The isomerization of 5,6-monoepoxycryptoxanthin to 5,8-monoepoxycryptoxanthin under acidic conditions (pH 3.5) would explain the hypsochromic shift of the total carotenoid extract in the puree and concentrate samples.

Provitamin A carotenoids in Solo papaya were reported to be  $\beta$ -carotene and cryptoxanthin, which comprise 4.8 and 38.9% of the total carotenoids, respectively (20). Cryptoxanthinmonoepoxide was reported to be 15.6%; it too, may be provitamin A depending on the position of the expoxide group. Since there is a question at this time on whether the epoxy group is 5,6 or 5',6', the provitamin A activity of this carotenoid is uncertain. However, the isomerization from the monoepoxy form to the furanoid form should not affect the provitamin A activity of the puree, because 5',6', epoxycryptoanthin would not affect the ( $\beta$ -ionone (provitamin A) portion of the molecule.

Total carotenoids decreased from an initial value of 2.83 mg% in fresh fruit to a final value after concentration of 2.12 mg%. Because of the hypsochromic effects, such losses in total carotenoid values should not be construed as destruction of carotenoids and provitamin A.

Changes During Storage. Frozen papaya puree that has been produced without the benefit of acidification and thermal inactivation of enzymes results in a smelly, gelatinized product. Gelation and off-flavor development occurs within a few months under frozen storage at 10°F. Puree packed into polylined 40 lb cartons for freezing will also exhibit

surface bleaching on the uppermost few millimeters of the bag, especially where the bag is twisted for sealing. This is due to the oxidation of the carotenoids.

b. Puree Manufacture. As stated previously, deleterious enzymatic activity is initiated in papayas whenever the fruit is pulped. These deleterious changes were subsequent gelling of the puree and development of off-odors and off-flavors. An improved method for producing papaya puree has been developed by the USDA-ARS and University of Hawaii-Manoa, which overcomes the enzymaticaly induced changes (21).

The papayas are first steamed for 2 min in a steam tunnel. Steaming the whole fruit before processing has the dual effect of preventing exudation of latex from the skin during slicing and of softening the outer 3 to 4 mm of the fruit, thus increasing the yields by 4 to 10%. Steaming the fruit also inactivates the enzymes in the outer 2 to 3 mm of the fruit. Steaming also serves to surface sterilize the fruit, thereby lowering the microbial load. The steamed fruit are then spray cooled on a conveyor, after which they are mechanically sliced through a set of circular blades set 1 to 1.5 inches apart. The sliced fruits are fed into a crusher-scraper device. Within this device the papaya slices are squeezed through a narrow gap and the flesh and seeds are loosened and removed from the peel by the action of a rapidly rotating cylinder. Breakage of seeds is minimized by the use of the crusher–scraper in contrast to the use of conventional pulpers. Minimization of breakage of seed and sarcostestae minimizes the release of the enzyme myrosinase and its substrate benzyl glucosinolate, thereby minimizing the development of off-flavors. The crushed flesh is then separated from skins and seeds in a centrifugal separator. The separation of skins and seeds by the centrifugal separator prior to pulping further minimizes the breakage of skins and seeds and their inclusion in the puree. The crushed flesh is then pulped in a paddle pulper fitted with a 0.033 inch screen. The pulped flesh is then acidified with citric acid to pH 3.5, which also inhibits the growth of microorganisms. The acidified puree is then pumped through the paddle finisher fitted with a 0.020 inch screen, which removes coarse fiber and whatever seed-coat particles are present, to yield a smooth puree. The puree is then pumped through a plate heat exchanger where the temperature is raised to 204°F, held for 2 min, and then lowered to 85°F, in a continuous flow. The heat treatment serves to inactivate the enzymes and destroy the microorganisms. The cooled puree is packaged into 40 lb containers and frozen at  $-10^{\circ}$ F.

The puree made by this method has proven to be superior to purees made by other methods. Purees manufactured by the new method are devoid of off-flavors and odors, do not gel during frozen storage, are lower in microbial counts, and possess fewer seed particles.

c. Frozen Papaya Piece-Form Products. Frozen papaya piece-form products generally experience a loss of texture, becoming soggy and mushy. Hence greener (one-half to three-quarters), firmer fruit are generally selected as the raw material for frozen papaya chunks or pieces. An assortment of frozen papaya products and freezing processes has been attempted with varying degrees of success. Papaya halves in which whole papayas were sliced and seeds removed have been frozen. This product has also been marketed with the seed cavity filled with scoops of ice cream or sherbet. Frozen papaya chunks and slices have also been manufactured and test marketed. The various freezing methods used were air blasting at  $-40^{\circ}$ C, immersion in a combination of sodium chloride and ethanol at  $-23^{\circ}$ C, smothering with a layer of flaked carbon dioxide, and drenching with cryogenics such as freon or liquid nitrogen.

A contributing factor to the limited commercialization of frozen papaya products is the lack of commercial equipment for deseeding and peeling. A mechanical means for removal of papaya seeds has been developed and patented (22). The principles of the seed removal process are based on the application of a fluid jet through an orifice in the papaya, thereby forcing the seeds out through an opening in the blossom end of the fruit. Various mechanical means for extracting papaya chunks has been developed. Papayas are sliced lengthwise into quarters, the seeds are removed, and the flesh side of the fruit is pressed down on a mesh belt. The mesh size determines the chunk size as the mesh whose upper surface acts as the cutting edge is either a sharpened edge or fine mesh stainless steel wire. The sliced fruit are then passed under a roller, which presses down on the fruit into the mesh. The chunks are then separated from the skin using a doctor blade. The fruit chunks usually tumble into a solution of calcium chloride, citric acid, and ethylenediaminete-traacetic acid.

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# **29** Frozen Citrus Juices

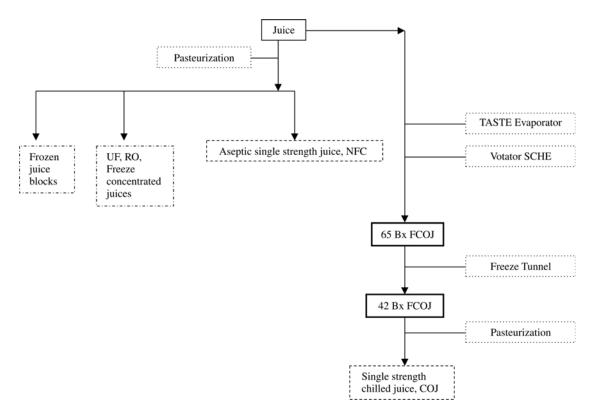
**Louise Wicker** 

University of Georgia, Athens, Georgia, U.S.A.

# I. HISTORY OF AND TRENDS IN THE CITRUS INDUSTRY

Citrus fruit processing concerns primarily frozen concentrated orange (FCOJ) and grapefruit concentrate. With the introduction of the TASTE evaporator, juice could be economically concentrated and stabilized. From the 1960s to the mid-1990s, freezing of orange juice concentrate was considered the best method of preserving citrus juice quality and flavor. The general consensus was that bulk storage and distribution of 65°Bx or 45°Bx concentrate provided a ready supply of consistent, high-quality citrus juice, and that single-strength juice could not be economically processed or distributed. Since the mid-1990s, processing of chilled juice not from concentrate (NFC) has steadily increased. A general process flow for the major juice products from frozen citrus concentrate and aseptically stored single-strength juice is depicted in Fig. 1. The two main products are single strength, not from concentrate (NFC) juice that is pasteurized and stored in chilled, aseptic storage for up to a year. The majority of juice is concentrated to 65°Brix in an evaporator, frozen with a scraped surface heat exchanger, and stored until either dilution to 42°Bx, or dilution to single-strength, repasteurized and distributed as chilled orange juice (COJ). Other nonthermal methods for concentration, such as ultrafiltration, reverse osmosis, or freeze concentration, are available but are of limited commercial use.

This chapter will cover some of the unit operations of citrus processing and the process parameters involved in making consistent high-quality frozen juices and juice concentrates. The focus and terminology will be on orange juice simply because the volume of this juice is so important. Unique aspects of processing and freezing of grapefruit juices will be introduced as needed. Interestingly, many of the processes of freezing citrus juices have not substantively changed in the last 40 years. From a historical perspective, the industry has responded to crises, and those responses provided for the development of diverse citrus juice products. Many of the crises faced by the citrus industry were precipitated by adverse climatic conditions. After the devastating freeze in the 1963 season, pulp wash was introduced as a means to salvage more solids from freeze damaged fruit (1). Quality attributes of citrus and definitions of quality were redefined in response to multiple freezes in the early 1980s (2). There have been process and product improvements for flavor, blending, stability, and processing efficiency. The target is to achieve a processed citrus juice for the consumer that captures the flavor and aroma notes



**Figure 1** Flow diagram of major juice products derived from citrus fruits. Reverse osmosis (RO), ultrafiltration (UF), not from concentrate (NFC), chilled oranged juice (COJ).

of fresh juice, irrespective of whether it originates from frozen concentrate or single-strength juice.

# II. CITRUS CULTIVARS, EXTRACTION, AND FINISHING OF JUICE

#### A. Citrus Cultivars

Citrus cultivars typically used for orange juice processing are the early season Hamlins, which mature in November and December, the mid-season pineapples, which mature in January and February, and the premium juice orange, Valencia, that mature from March to June. In the orange cultivars, variations in color score, solids content, and acidity are monitored during processing, and juices and juice concentrates are blended to achieve desired color and °Br–acid ratio. Hamlins tend to be lower in solids content and have lower color score. The pineapple orange has excellent color and flavor scores but is subject to variable bearing. Valencia orange juice is among the highest scoring juices in terms of color, °Bx–acid, and flavor. In the absence of freeze years, the late maturing Valencia orange provides the base juice for blending to achieve consistent high-quality juice.

Grapefruit varieties include Duncan, Marsh, and Ruby Red. Marsh grapefruit have fewer seeds and are the most common grapefruit harvested in Florida. Ruby and Star Ruby have a pink to dark pink blush, respectively (3).

# B. Citrus Harvesting

Citrus fruit are harvested either by hand or by machine and transported to the factory by tractor-trailer trucks. The fruit is unloaded, and air/water sprays remove trash, stems, leaves, and twigs. Fruit are graded to remove unwholesome, moldy, or bruised fruit and stored in covered storage bins in a metal mesh frame structure. The bottoms of the bins are slanted to minimize bruising due to pressure and to facilitate discharge of the fruit. After removal from the fruit bins, the fruit are washed with detergent water sprays and rinsed with 10–20 ppm chlorine. Fruit are graded and sized by parallel conveyors.

#### C. Juice Extraction

Two types of extractors are commonly used, the Brown extractor and the FMC extractor. The Brown extractor slices fruit in half and extracts juice by reaming the fruit. A hard or soft squeeze may be used depending on end-product use. Higher pressures result in higher yields but also higher contents of limonene, pectinesterase, pulp, and pectin. Lower pressure extractions result in better flavor and color scores. A hard or soft squeeze has a clearance of about 0.16 cm or 1.11 cm between the cup and the wall of the fruit, respectively (4). In the FMC extractor, the fruit falls into finger like cups. Top and bottom plugs are cut in the fruit, and high pressure forces juice into a perforated stainless steel prefinishing tube. The peel is discharged between the upper cup and the cutter. At the end of the pressure increase, an orifice tube moves upward and increases pressure in the prefinisher tube. Juice and juice sacs flow through the holes of the prefinisher tube, and large pieces of peel, pulp, and seeds are discharged through the bottom of the orifice tube.

#### D. Heat Pasteurization

Citrus juice is heat pasteurized to inactivate pectinesterase (PE), the enzyme responsible for cloud loss in citrus juices and gelation of citrus concentrates. The most commonly accepted theory for clarification is that PE de-esterifies the methoxyl ester of pectin, and the resultant carboxylic acid can bind calcium or other cations to form large, unstable complexes. These calcium pectate complexes occlude other cloud constituents and result in a clear serum and an opaque layer in the sediment. In concentrates, pectic acid may form gels with calcium that do not reconstitute to a homogenous dispersion upon stirring. In severe gelation, the concentrate cannot be poured from the container. Pasteurization of citrus juice through plate heat exchangers or tubular heat exchangers is most common for juices that are designated for aseptic processing and for juices that are reprocessed after dilution and blending. For juice that will be evaporated, juice is pasteurized in the first stage of the evaporator. The time-temperatures required for heat inactivation for PE were established by Eagerman and Rouse (5). Versteeg et al. (6) showed that thermal pasteurization of citrus juice was based on inactivation of a thermostable PE that represented less than 5% of the total activity, and clarification was attributed to this isozyme. More recently, it was shown that thermolabile PE also clarifies juice (7), and other proteins influence the rate of clarification (8). Hence undesirable clarification of citrus juice probably results from multiple enzymatic reactions and interactions of cloud constituents. To decrease the thermal treatment, some processors take advantage of the heat sensitivity of PE to lower pH in pasteurization of early season fruit. The pasteurization process remains essentially unchanged, in spite of a plethora of literature that provides various inactivation data, heat stability, and characteristics of various PE

isozymes. Typical industry practices include protocols of 0.8 to 1 min at 90°C for a 99% inactivation of PE in the TASTE evaporator. Severe heat treatment, which is detrimental to the fresh flavor of citrus juice, could be avoided if the process temperature could be reduced to 70°C. Destruction of pathogens and spoilage organisms occurs at temperatures about 20°C lower than needed to inactivate PE. Some of the proposed regulations regarding the microbial pasteurization of reconstituted juice are undergoing review, but, generally the high acidity, low water activity, and cold temperatures that are necessary to preserve the quality of juice minimize pathogen problems.

# III. PRODUCTION OF FROZEN CONCENTRATE

# A. The TASTE Evaporator

In the early 1960s, low-temperature evaporators were replaced with TASTE (thermally accelerated short time evaporator) evaporators. Steam and vapor recovered from one stage is used to heat the next stage. Each step down in steam temperature is termed an effect. Contemporary TASTE evaporators will have up to eight stages and as many effects as stages. Product is concentrated as the stage number increases, and process temperatures decline with each increase in effect number. Compared to low-temperature evaporators, TASTE evaporators reduced process time from hours to less than 10 min (9, 10), removing 80,000-200,000 pounds (36,000-91,000 kg) of water per hour. Juices are rapidly heated to 93–112°C (9) in one of the early stages of the evaporator, which inactivates pectinesterase and microorganisms. Originally, external heat was applied in the first stage of the evaporator, but essence quality is improved if external heat is applied to partially concentrated juice in a later stage. Heat from this juice is used to heat fresh juice entering the evaporator. In each stage, bundles of vertical tubes are heated in an enclosed shell with vapors from the preceding stage. A vacuum is maintained in the tube, and juices fall under turbulent flow to the bottom of the tube. Water is flashed off as heated juice falls through vertical tube bundles. In addition to concentrating the juice, flashing cools the product and removes most oxygen and flavor volatiles. The volatiles are collected in an essence recovery unit and eventually added back to the product. Partially concentrated juice is pumped to the next stage, and the process is repeated. The TASTE evaporator produces a bland concentrate of about 65°Bx that is called "pumpout." A small amount of peel oil is added back to prevent the development of a cardboard off-flavor.

# B. Freezing in a Scraped Surface Heat Exchanger

Juice concentrates from the TASTE evaporator are rapidly chilled in a scraped surface heat exchanger. The Votator II (Waukesha Cherry Burrell, Louisville, KY) is commonly used (Fig. 2). A thin stream of concentrate (pumpout) is pumped between the space of the heat transfer tube and the center shaft. The shaft has staggered rows of scraper blades (Fig. 2) that remove the frozen film, and the concentrate exits the Votator at temperatures near  $-7^{\circ}$ C. The frozen slush is typically pumped into 50,000-250,000 gallon tank farms and stored at  $-9^{\circ}$ C under a nitrogen flush for up to a year. It has been reported that some tank farms have a capacity of 20 million gallons of concentrate (1). The  $65^{\circ}$ Bx is diluted to  $42^{\circ}$ Bx for distribution as FCOJ and blended prior to redistribution. If packed into retail cans, the blended  $42^{\circ}$ Bx FCOJ is rechilled through the Votator prior to entering the filling machine; it is then air-blast frozen at  $-30^{\circ}$ C (see Fig. 3).



Figure 2 Votator (Waukesha Cherry Burrell) scraped surface heat exchanger to freeze FCOJ.

In general, process design is based on the fluid flow properties of the concentrate (11). Freezing and blending of citrus juice is often limited by the capabilities of the filler. Single-strength juice cannot reasonably be chilled below -2 or  $-3^{\circ}$ C because it cannot be pumped. Low moisture pulp also cannot be used in-line, because the filler cannot handle the pulp without added juice.

#### C. Citrus Flavors and Essences

The flavor and aroma of fresh juice is difficult to mimic and is typically attributed to aldehydes, esters, ketones, alcohols, and hydrocarbons. Sources of citrus flavors are the cold-pressed oils recovered during extraction as well as essences recovered during concentration. An essence recovery unit is a series of fractionating condensers (12). The aqueous essence is the volatile water-soluble product recovered during concentration and



**Figure 3** Freezing tunnel to freeze individual packages of 42°Bx FCOJ.

is 7–18 times higher in ester content than cold-pressed oils (13). Oil essence is decanted from the aqueous essence and is approximately 85% D-limonene. The remainder is a mix of esters, aldehydes, ketones, alcohols, and hydrocarbons. The bouquet and flavor of fresh juice is closely mimicked by addition of essence. Typical use levels are about 0.20% aqueous essence and 0.01% oil essence (12).

Preserving fresh juice flavor in heated juice is difficult. In FCOJ, fresh flavor is mimicked by adding back orange oil and essences that were stripped in the evaporation process. Through the 1960s and 1970s, it was common for the industry to add a small amount of unheated juice to FCOJ as "cut back" juice. Periodic problems resulting from inadequate heat inactivation of PE, excessive additions of fresh juice, as well as temperature abuse during distribution and handling ended the practice. Cut back juice was discontinued in the mid to late 1980s as a method to enhance flavor. Fellers et al. (2) reported that quality and consumer acceptance of FCOJ were lower if freeze-damaged oranges were used to make FCOJ. With changes in regulations including the lowering of the minimum "Bx of concentrate and single-strength juice, they evaluated the interrelationship between physical and chemical characteristics of FCOJ and consumer sensory scores. In general, consumer preference was associated with higher "Bx—acid ratios. Blending juices for flavor and quality incorporates several techniques. Seasonal variation in addition to different production and processing practices results in juices of varying quality and flavor. Blending of juices in industry based on knowledge of

physicochemical characteristics requires technical know-how and artful experience (14). Chen (15) reported a nonlinear relationship between flavor scores and °Bx–acid ratio, and the optimum ratio was not necessarily the highest ratio. Flavor scores increased with increasing °Bx–acid ratio to about 8.5 and remained constant until a decline at °Bx–acid ratios greater than 10. He presented a mathematical model to be used in blending juice for optimum flavor scores that provides a logical basis for blends of high and low ratio juices for optimum flavor.

# D. Chilled Orange Juice from Concentrate (COJ)

The most convenient source of chilled orange juice is diluted and repasteurized orange juice concentrate. FCOJ tank farms ensure a steady supply and blending flexibility for juices of optimal quality. Reconstituted juices must be repasteurized and labeled with "From Concentrate" on cartons.

# E. Grapefruit

Frozen grapefruit concentrate is processed by virtually the same technology as for orange concentrate. The time-temperature profile to inactivate PE is typically lower for grapefruit, and pulp content is lower, near  $85^{\circ}$ C,  $z = 6^{\circ}$ C (1).

#### F. Shelf Life of FCOJ and COJ

The shelf life of pasteurized citrus juice concentrates is primarily limited by chemical reactions. Most microbial stability problems are controlled by the thermal concentration processes that are sufficient to inactivate degradative enzymes such as thermostable pectinesterase. Process control and storage for microbial stability is easier in juice concentrates than in single-strength juice (16). The lower water activity (0.80-0.83) precludes microbial growth in most cases, and the high solids content allows storage at low temperatures (about  $-5^{\circ}$ C to  $-9^{\circ}$ C). The major spoilage bacteria that would limit the shelf life of citrus juice concentrates are *Lactobacillus* and *Leuconostoc*, which are responsible for buttery off-flavor. Species of *Saccharomyces* and *Candida* are fermentative yeasts that contribute to fermented off-flavors (16). Citrus concentrates should be monitored for heat resistant molds, *Byssochlamys* and *Penicillium* spp., that may grow in packaged products with sufficient oxygen (17). Minimal changes in microbial stability are reported for deaerated FCOJ stored at -5 to  $-9^{\circ}$ C under a nitrogen flush.

The loss of citrus juice and concentrate quality as well as short shelf life results from multiple chemical reactions involving several substrates including ascorbic acid, essential oils, lipids, phenolics, and sugars (17,18). Thermal processing, prolonged storage, exposure to oxygen, and acidity contribute to the loss of flavor and the development of off-flavor. Oxidation of ascorbic acid proceeds by multiple pathways, with the formation of several intermediate and end products. In addition, anaerobic degradation of ascorbic acid also leads to the formation of furfural and brown pigments. The complexity of aerobic or anaerobic degradation of ascorbic acid has made prediction of quality degradation difficult.

Nevertheless, furfural, which derives from ascorbic acid degradation and other degradative reactions with sugars and amino acids, is an accurate index of thermal abuse and the development of off-flavors (19). The accumulation of furfural is greater in single-strength juices than in concentrates. The accumulation of furfural in concentrates is

directly related to higher temperatures and indirectly related to solids content (17). When  $66^{\circ}$ Bx orange concentrate was stored between  $-18^{\circ}$ C and  $+4.4^{\circ}$ C for 0 to 12 months, nonenzymatic browning increased and sensory scores decreased with higher temperatures and longer times (20).

Oxidative degradation may occur from oxygen in the container headspace, from that entrained in the product, or from permeation through the container, but oxidation is less of a problem in concentrates than in fresh or pasteurized juices (17). Headspace volumes of 20% did not affect quality of 60°Bx concentrates that were stored at 0–2°C for up to 10 months. The method of deaeration, by vacuum, nitrogen sparging, or hot filling of concentrates, did not result in significant differences in quality or shelf life of concentrated grapefruit juice. Storage temperature was the most important parameter in the shelf life of aseptic citrus juice and concentrates (17). Minimal changes in microbial, chemical, and flavor stability were reported when deaerated FCOJ contained in stainless-steel tank farms was stored at low temperatures under a nitrogen flush.

Stability and quality become more critical when FCOJ is diluted to threefold concentrates or to single-strength juices, oils and essences are added, and it is packaged for distribution. FCOJ may be repackaged into threefold concentrate for retail distribution, 100 gallon bag-in-box aseptic packaging, 55-gallon stainless steel drums with polyethylene liner, or single strength juice packed in cartons. The aseptic cartons and liners used will vary but will typically have polyethylene or polypropylene in contact with the juice and a metallized laminate as a tie layer between inner and outer polyethylene. Oxygen, storage temperatures, and packaging materials are major factors in quality. Single-strength juices, whether from concentrate or not from concentrate, are similar in susceptibility to flavor loss, ascorbic acid degradation, and other degradative reactions.

Packaging and transport influence quality, and high oxygen barrier films are needed that do not affect the flavor quality of single-strength juice within an expected shelf life of about 90 days. In an evaluation of different polymers, single-strength juice from concentrate with added orange oil was mixed with different polymer beads (21). Within 4 days equilibration, absorption of orange oil was highest in low-density polyethylene, intermediate in suran, and lowest in high-density polyethylene and polypropylene. Limone and other terpenes were adsorbed at greater than 50%. Octylacetate ester was also highly adsorbed at 34-40% and longer chain length aldehydes were adsorbed more than shorter chain length. Loss of quality of aseptically packed single-strength juice from concentrate was more rapid when packed in low-density polyethylene (LDPE) than glass and stored at 25°C or 35°C (22). Greater browning, loss of ascorbic acid, and loss of D-limonene was attributed to the adsorption to LDPE and transmission of oxygen through the package. Sensory quality was less after 10–12 weeks of storage in the LDPE packed juices (22). The shelf life of orange juice and grapefruit juice concentrate packed in aseptic bag-in-box packages was reduced by residual oxygen and oxygen permeation from the spout and laminate film (23). Minor differences were observed between laminate films (Cotlab, Liquibox, BXL). Storage life was more affected by storage temperature, and greater loss was observed at 25°C than 4°C in ascorbic acid, and browning increased (23). With similar degradative mechanisms in reconstituted juices and not-from-concentrate juices, major changes in quality can be expected with higher storage temperatures and oxygen concentrations in stored single-strength juices. In a study on aseptic juice, Moshonas and Shaw (24) reported decreases in volatiles and large changes in water-soluble constituents after only 2 days at 2°C. Sensory results suggested no detectable changes through the 9 week study. Marcotte et al. (18) took a somewhat different approach by evaluating the effects of temperature, pH, and oxygen on the production of degradative by-products that

result in stale, musty off-flavors,  $\alpha$ -terpineol, and  $\rho$ -vinylguaiacol. They reported that the most effective way to prevent off-flavors was to reduce dissolved oxygen. Longer shelf life juices will likely require better barrier packaging materials, low residual oxygen, and constant low temperatures.

In stored citrus juice concentrates, oxidative reactions and thermal abuse are the major factors contributing to the decline of nutritional quality, color, and flavor of concentrates and single-strength juices from concentrate. Deaeration, nitrogen purging, the use of oxygen scavengers in packaging materials, oxygen barrier packaging, oxygen barrier in packaging spout, and good temperature control throughout storage and distribution are essential components of extended shelf life juices.

# IV. PULP AND PULP WASH CONCENTRATE

After finishing of juice, the residual juice sacs and pulp contains residual juice solids that can be recovered as pulp wash or can be heat stabilized and frozen for use as other byproducts. The pulp washing process balances the recovery of further juice solids and high viscosity due to pectin.

Addition of pulp wash to juice was not allowed in the U.S. until the mid-1990s when the FDA decreed that the addition of in-line pulp wash to make juice concentrates did not violate the standards of identity for citrus juices. Pulp wash concentrate with enzymatically reduced viscosity can be processed in an almost identical process scheme to that for first-run juices for FCOJ, but addition to juice is not permitted. Pulp wash concentrates are used in lower quality juice products and juice blends.

The juice sacs that are not washed to recover solids may be added back to juices and juice beverages to simulate fresh squeezed home-style juices. Floating pulp, free of defects, is desirable. An interesting method of monitoring defects of juice sacs was developed in the quality assurance program at Pasco Beverages (personal communication). Pulp sacs are placed in a petri plate with standard plate count agar and allowed to set. The result is a three-dimensional view of pulp sacs and shape. The integrity and typical canoe shape of undamaged juice sacs is easily visualized by this method.

High-pulp homestyle juices are popular with some consumers. In processing this product, heat stabilized pulp is added to either reconstituted or chilled single-strength juice at 25 g/L to 30 g/L depending on the processor. The pulp must be added so that the processor does not dilute the concentrate below target. Pulp to be added to juice must be PME negative; therefore it receives a heat treatment in a scraped surface heat exchanger and is either packed into 300 gallon aseptic Scholle bags or frozen in 5 gallon bags or 55 gallon drums. For pulp to be filled into Scholle bags, it must be diluted with about two parts of juice to keep the Scholle filler free flowing. This dilution of pulp with juice adds an additional level of control, since FCOJ must be diluted to a higher °Bx, approximately 49°Bx, to compensate for the higher moisture pulp. If pulp for homestyle juice is to be added directly, it should be very dry pulp that receives a hard finish in the finisher after extraction. Screens on the finisher are set at 0.02 inches to remove excess moisture/juice from the pulp. Freezing pulp may be as simple a procedure as filling a plastic-lined 55 gallon drum and placing it in a freezer. Braddock (1) estimated that juice sacs would freeze in about the same time and with the same energy costs as frozen citrus sections. He reported that previous estimates of sections in a 55 gallon drum of grapefruit sections would require 5 days to freeze from ambient to  $-22^{\circ}$ C. The actual time to freeze a 55 gallon drum is probably 10 days. Low moisture pulp is more typically frozen in 5 gallon boxes in an air blast ( $\sim 60 \, \mathrm{mph}$ ) freeze tunnel. Filled boxes are stacked on pallets with 12 cases to a pallet. A 3 inch buffer between pallets allows airflow, and pallets may be stacked as high as 5 tiers. The target temperature of  $-22^{\circ}\mathrm{C}$  is achieved in about 6 hours. A continuous method of prefreezing or slush freezing pulp without added moisture is highly desirable, but technologically it is not possible with scraped surface heat exchangers. Water must be added to the pulp in order to slush freeze it, and the solids content is so low that the freezer would tend to plug during processing.

# V. COLLIGATIVE PROPERTIES

Like any food undergoing freezing, citrus juices undergo changes in the ice water phase, temperature, latent heat of fusion, specific heat, enthalpy, and water activity. Information on physical properties of citrus juices aids in the prediction of freezing properties, the calculations of energy that must be removed to freeze water in juice, decrease the temperature, and give information on the amount of water that is unfrozen. Thermophysical properties may be directly measured or mathematically estimated. However, physical properties of foods are difficult to measure below freezing temperatures and are often estimated from extrapolations of properties of ice and water. Studies on the properties of ice water are valid, since changes in frozen foods are associated with the unfrozen water phase. The amount and properties of supercooled water influence the mechanism of physical and chemical changes in frozen food systems (25). In an elegant treatment of the thermodynamic properties of ice water at below freezing temperatures, Chen (26) presented a model for the systematic calculation of water activity, molal heat of fusion, and specific heat of supercooled water and used this to estimate further the thermodynamic properties of frozen food systems (Table 1). The table presents data of calculated properties of an ice water system as a function of temperature using the following equation:

$$A_{\rm w} = \frac{P_{\rm i}}{P_{\rm w}} = \frac{1}{1 + 0.0097\Delta T + C\Delta T^2}$$

where  $P_{\rm i}$  and  $P_{\rm w}$  are vapor pressures of ice and supercooled water and  $C=5\times 10^{-5}\,K^{-2}$  based on equilibrium vapor pressures of ice and water. Good agreement between calculated and experimental parameters was noted. This information is useful for understanding the mechanisms of physical and chemical changes in foods in the unfrozen water phase. Information on the amount and thermophysical properties of supercooled water will aid in this understanding.

Chen (27) derived equations for the use of freezing point depression to estimate enthalpy and apparent specific heat for citrus juice at 10–50% solids content and at temperatures between  $20^{\circ}$ C and  $-30^{\circ}$ C. His calculated values were in close agreement with experimental data and could be used as a basis for the estimation of physical properties at other temperatures and for the prediction of the rate of ice formation at various temperatures (Table 2). In subsequent work, Chen (28) calculated enthalpy and apparent specific heat and ice content based on freezing point depression (FPD) and reported high accuracy with experimental values in citrus juice. The FPD estimation of enthalpy and apparent specific heat was applicable over a broad temperature range to citrus juice. He also estimated the amount of bound water at  $-40^{\circ}$ C using Schwartzberg's method and found good agreement depending on the accuracy of the initial freezing point

**Table 1** Calculated Properties of an Ice–Water System at Temperatures from 273.15 to 233.15 K

<i>T</i> , K	Δ <i>T</i> , K	$A_{ m w}$	$\underline{L}_{\mathrm{T}}$ , kJ/kg	$L_{\rm T},~{\rm kJ/kg}$	$\Delta$ $C_{\rm p}$ , kJ/kg K	$C_{\rm pi},~{\rm kJ/kg}~{\rm K}$	$C_{\rm pw}$ , kJ/kg K
273.15	0	1.00	334.20	334.20	2.13	2.06	4.19
270.15	3	0.971	330.93	327.81	2.13	2.06	4.19
268.15	5	0.953	328.81	323.46	2.20	2.03	4.23
265.15	8	0.925	325.52	316.62	2.32	2.01	4.33
263.15	10	0.907	323.25	311.88	2.40	2.00	4.40
260.15	13	0.881	319.77	304.52	2.49	1.98	4.47
258.15	15	0.864	317.39	299.46	2.54	1.97	4.51
255.15	18	0.839	313.74	291.69	2.62	1.95	4.57
253.15	20	0.823	311.26	286.38	2.66	1.94	4.60
250.15	23	0.799	307.48	278.27	2.73	1.92	4.65
248.15	25	0.784	304.92	272.78	2.76	1.91	4.67
245.15	28	0.761	301.02	264.42	2.79	1.89	4.69
243.15	30	0.747	298.38	258.78	2.83	1.88	4.71
240.1	33	0.725	294.39	250.24	2.85	1.86	4.71
238.15	35	0.712	291.69	244.50	2.87	1.85	4.72
235.15	38	0.692	287.62	235.84	2.90	1.83	4.73
233.15	40	0.679	284.88	230.04	2.90	1.82	4.72

T, K = temperature;  $A_{\rm w}$  = water activity;  $\underline{L}_{\rm T}$ , kJ/kg = average molal latent heat of fusion of ice;  $L_{\rm T}$ , kJ/kg = molal latent heat of fusion of ice at T;  $\Delta C_{\rm p}$ , kJ/kg K = difference in specific heat of water and ice;  $C_{\rm pi}$ , kJ/kg K = specific heat of ice;  $C_{\rm pw}$ , kJ/kg K = specific heat of supercooled water. Source: From Ref. 26.

measurement. Freezing point depression can be reasonably measured, and Chen and Nagy (29) used solvation theory to predict constants for solute—water interactions. They observed a nonlinear power law response of concentration to freezing point depression. Chen (30) used FPD to estimate water activity of different foods, including citrus juices (Table 3). Estimation of water activity has a variety of applications with respect to the prediction of chemical and oxidative changes in the quality and flavor of citrus juices.

**Table 2** Calculated vs. Experimental Enthalpy Values for Orange Juice with 89% Moisture Content (Reference Temperature 20°C)

	Enthalpy (Kcal/kg)			
Temperature, °C	Experimental (from Riedel)	Calculated (from Chen, 1985)		
-29.92	101.5	100.3		
-19.65	95.0	94.7		
-14.50	91.2	91.1		
-9.34	85.7	85.9		
-4.17	73.0	72.7		
-0.05	18.7	18.8		
20	0	0		

Enthalpy is 0 at 20°C. *Source*: From Ref. 27.

**Table 3** Comparison Between Experimental and Calculated  $A_{\rm w}$  Values for Citrus Juice Concentrates

Fruit concentrate	°Bx	$A_{\rm w}$ : experimental	$A_{\rm w}$ : calculated $M_{\rm s} = 240$	$A_{\rm w}$ : calculated $M_{\rm s} = 202$
Orange	15	0.982	0.986	0.984
-	40	0.908	0.945	0.936
	60	0.84 – 0.87	0.861	0.840
	65	0.802 - 0.835	0.818	0.791
	72	0.735	0.716	0.679
Grapefruit	59	0.844 – 0.886		0.847
Lemon	40–42	0.90 – 0.932	_	0.936-0.930

 $M_{\rm s}$  is molecular weight of solute. Source Adapted from Ref. 30.

Further, information on water activity at different °Bx is useful to confirm the need for thermal inactivation by pasteurization of concentrates to be stored in tank farms.

Experimental freezing point depression in concentrates with greater than about 25% by weight did not follow FPDs estimated by equilibrium freezing curves (25). Chen attributed the deviation to water binding properties of the solutes. Chen et al. (31) reported that sugars and acids were not the solutes responsible for the deviation in FPDs estimated from equilibrium freezing curves but did not offer suggestions as to other possible constituents in juice, such as pectins and proteins, that could account for water binding.

# VI. FREEZE CONCENTRATION

Freeze concentration was popular in the Florida juice industry in the late 1980s and 1990s. A major equipment manufacturer is the Grenco freeze concentrator. Freeze concentration of liquid foods involves the crystallization of water to ice and then selective separation of the ice crystals (32). Careful preprocess treatment is more critical than for evaporative processes, but freeze-concentrated juices retain more aroma constituents associated with fresh juice because of the lighter heat treatment (33, 34). The three basic components of a freeze concentrator are the heat exchanger, recrystallizer, and wash column (35). Citrus juice is fed from a surge tank and frozen in a scraped surface heat exchanger to form small ice crystals. The ice crystals are then pumped to the recrystallizer, where the small crystals are mixed with larger crystals. Because of the slightly lower equilibrium temperature of small than that of large ice crystals, the smaller ice crystals will melt and recrystallize onto the larger crystals. Wash columns achieve separation of the ice crystals from the concentrate, which concentrate is removed through the bottom of the wash column leaving a layer of ice crystals at the top. The ice is then scraped away, melted, and used to wash the packed bed. A clear separation of concentrate and ice crystals—wash water will form that is termed the wash front. The limit of concentration for freeze concentration is lower than for evaporative methods, near 50°Bx. The potential of freeze concentration has not yet been fully realized. The technology is sound, and there is no doubt that it forms a superior concentrated product. However, high capital costs, equipment maintenance, loss of juice solids, and the limit of concentration have restricted its widespread application. In addition, feed juice must still be pasteurized prior to freeze concentration.

# VII. FROZEN JUICE BLOCKS

Frozen juice blocks were the first practical long-term storage source of single-strength juice of premium quality (14). Pasteurized single-strength juice is flash frozen in blocks, frozen in blast tunnels at approximately  $-18^{\circ}$ C, and used for blend stock in FCOJ. Blocks are crushed, melted, blended with other juices as needed, pasteurized, and filled into retail containers. Frozen blocks offer the same blending and year-round retrieval benefits of the FCOJ tank farm. The use of oils and essences has diminished the relevance of this technology, and the rise in aseptic chilled juice has further depreciated its application. However, the need for more efficient freezing of stabilized pulp could be met by the adaptation of freezing single-strength blocks of juice to freezing of blocks of stabilized pulp.

#### VIII. ASEPTIC BULK STORAGE

Unlike many fruit juices, citrus juices experience severe quality loss 6 weeks after aseptic processing when stored at ambient temperatures. Refrigerated aseptic bulk storage vessels made aseptic processing practical for the citrus juice industry by increasing the storage life to over 1 year at -2°C. Aseptic bulk storage vessels contain 250,000–500,000 gallons of juice. Steam or chemical treatments are used to sterilize aseptic tanks. Fresh juices are immediately pasteurized, chilled, and pumped into aseptic storage tanks. The tanks are continuously purged with nitrogen to suspend the pulp and minimize oxidative reactions. After blending with other juice streams, aseptic juice receives a second thermal treatment. Flavor volatiles may be stripped away during nitrogen purging, and as a result aseptic juice is considered to be of lower quality than frozen block juice. Aseptic juice may be blended with flavor oils and essences prior to retail packaging.

# IX. USE OF ENZYMES IN CONCENTRATE PRODUCTION

Although pectic and cellulytic enzymes are not allowed as processing aids in citrus juice, interest in their use persists. Enzymes reduce viscosity and improve process efficiency of pulp wash concentrates. Certainly, enzymes are excellent process aids in concentrated juices, and they reduce viscosity and allow for concentration to higher °Bx. Until the 1990s, bulk storage of SSJ was deemed economically unfeasible, and process methods to produce higher °Bx concentrates were thought to be economically sound, considering that no practical loss of quality was measured in FCOJ as a result of enzyme use to reduce viscosity (36). Some reports indicate that the use of enzymes can actually provide juices of superior color and cloud stability (37). Consumer perceptions that not-from-concentrate juices are higher in quality have driven the aseptic juice industry. Research in the area of enzymes as process aids would have potential application beyond the 100% citrus juice markets and provide improved process efficiency and control for processing of juices for citrus juice blends, citrus-juice-containing beverages, and citrus-based drinks.

There is considerable interest in better prediction and control of process streams to make the best product with minimal energy use. Process developments such as reverse osmosis (RO), ultrafiltration (UF), and freeze concentration (FC) have lesser effect on flavor than thermal effects of the evaporator, but they are energy intensive, have high

capital costs, or produce low yields. A pasteurization step is still needed, so thermal effects are not completely eliminated.

# X. CONCLUSIONS

The popularity of convenient single-strength juice from concentrate (COJ) is high and accounts for a large portion of the use for FCOJ at the consumer level. The consumption in aseptic processing and bulk storage of chilled single-strength juice (NFC) will likely remain in the future of citrus juice processing. At the present time, the volume of FCOJ is greater than the volume of NFC, but the latter is increasing. Research and industry practices have come close to achieving the exquisite flavor and aroma of fresh squeezed juice. However, because high-temperature pasteurization is needed to inactivate detrimental enzymes, thermal deterioration of flavor still results. Nevertheless, the industry, with a commitment to quality, provides a consistent supply of premium quality juice from frozen concentrates.

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# **30**

# **Ice Cream and Frozen Desserts**

# H. Douglas Goff

University of Guelph, Guelph, Ontario, Canada

# Richard W. Hartel

University of Wisconsin-Madison, Madison, Wisconsin, U.S.A.

This chapter is focused on frozen desserts, dairy or nondairy, that are characterized by being concomitantly whipped and frozen in a scraped surface freezer and subsequently consumed in the frozen state. There are many product variations on this category, ice cream and lower fat versions being the most common, but also including sherbets and sorbets, frozen yogurt, soy-based frozen desserts, etc. Thus we begin with definitions and formulations of the major products within this category. However, there are many features of these products that are similar, hence many other aspects can be treated collectively. We will review the sources and functional roles of ingredients, mix manufacturing, including formulation calculations, the dynamic freezing process, including structure and structure formation, the static freezing (hardening) process, product storage and distribution, and finally, a review of shelf life and quality aspects. Although we use "ice cream" in the generic sense throughout this chapter, all of these topics are relevant to all products within this category.

It is not possible to provide a complete coverage of all aspects of ice cream and frozen desserts in one chapter. However, various aspects are covered in numerous books (1,2), book chapters (3–8), and review papers (9–11).

#### I. FORMULATIONS AND INGREDIENTS

#### A. Product Definitions and Formulations

#### 1. Ice Cream

The most common product within the category of frozen desserts is ice cream. The legal definition of ice cream is controlled by regulations and varies with jurisdiction, but it is generally a sweetened product containing milk fat and milk solids-notfat (msnf) and is frozen while being whipped. The general composition of most ice cream products is shown in Table 1.

Some of the factors affecting the choice of composition include legal requirements, which must be met, the quality desired in the finished product (increasing fat and total solids are usually associated with increasing quality), and the cost to be borne by the

**Table 1** The General Composition of an Ice Cream Mix

Component	Range of concentration
Milk fat	> 10%-16%
Milk solids-not-fat	9%-12%
Proteins, lactose, minerals	
Sweeteners	
Sucrose	10% - 14%
Corn syrup solids	3%-5%
Stabilizers	0%– $0.25%$
Guar, locust bean gum (carob), carrageenan,	
carboxymethyl cellulose (cellulose gum),	
microcrystalline cellulose (cellulose gel),	
sodium alginate, xanthan, gelatin	
Emulsifiers	0% - 0.25%
Mono- and di-glycerides, Polysorbate 80	
Water	55%-64%

consumer. Premium products usually command a higher price. There are no specific definitions of common industry-accepted terms such as premium or superpremium ice cream, but a relationship between fat content, total solids content, air content, and cost (also affected by quality and proportion of inclusions and marketing issues) exists, as illustrated in Table 2.

Suggested formulations for a range of ice cream products are presented in Table 3. Several trends are evident. There is usually an inverse relationship between fat and total solids compared to msnf. As discussed in Sec. B.2, the lactose component of the msnf is quite insoluble and above its saturation level in ice cream, so with increasing lactose content in a decreasing quantity of water, the risk of lactose crystallization increases. There is also generally an inverse relationship between corn syrup solids (starch hydrolysate sweetener, sometimes referred to as "glucose solids") levels and total solids. The corn syrup solids will contribute to a firmer, chewier texture, which is more desirable when there are less solids present. Likewise, as total solids increases, there is less requirement for stabilizer. This is generally because increasing stabilizer-in-water ratios lead to enhanced guminess, which becomes undesirable at high levels, and also a reduction in the water content means there are diminished problems associated with ice

**Table 2** Average Values for Fat and Total Solids Contents, Overrun and Cost Among the Categories of Ice Cream

Component	Economy	Standard	Premium	Superpremium
Fat content	Legal minimum, usually 10%	10–12%	12–15%	15–18%
Total solids	Legal minimum, usually 36%	36–38%	38–40%	> 40%
Overrun Cost	Legal maximum Low	~ 100% Average	60–90% Higher than average	25–50% High

**Table 3** Suggested Mixes for Hard-Frozen Ice Cream Products (%)

Milk fat	10.0	11.0	12.0	13.0	14.0	15.0	16.0
Milk solids-not-fat	11.0	11.0	10.5	10.5	10.0	10.0	9.5
Sucrose	10.0	10.0	12.0	14.0	14.0	15.0	15.0
Corn syrup solids	5.0	5.0	4.0	3.0	3.0	_	_
Stabilizer <sup>a</sup>	0.35	0.35	0.30	0.30	0.25	0.20	0.15
Emulsifier <sup>a</sup>	0.15	0.15	0.15	0.14	0.13	0.12	0.10
Total solids	36.5	37.5	38.95	40.94	41.38	40.32	40.75

<sup>&</sup>lt;sup>a</sup>Highly variable depending on type; manufacturers' recommendations are usually followed.

recrystallization. Also, as fat levels in a mix increase, there is generally less need for emulsifier, in order to optimize the extent of partial coalescence of the fat. Further discussion on many of these aspects of formulations can be found in the appropriate sections of the chapter.

Soft-serve ice cream is very similar to its hard-frozen counterpart in composition, but it is sold at a different point in its production stage, and usually with a much lower overrun content. Suggested formulations are shown in Table 4 for soft-serve ice cream, but it should also be recognized that much of the soft-serve on the market today falls into the low-fat or ice milk category, with fat contents typically around 4%. Generally, while the fat content is kept lower, the msnf content is higher than for hard-frozen products. Lactose crystallization is not a problem in these products, as they are consumed immediately after freezing. Corn syrup solids are often used but can lead to an enhanced sensation of guminess. Stabilizers are also generally used for viscosity enhancement and mouth feel, but their function in ice recrystallization is no longer needed. Dryness and shape retention, however, is a big concern in soft-serve products, hence the emulsifier content is generally kept high.

# 2. Reduced Fat Products

Ice milk was the traditional lower fat ice cream product for many years, but this category has been reclassified by many regulatory jurisdictions to include three reduced fat categories: light ice cream, low-fat ice cream (the traditional ice milk), and nonfat ice cream. Light or "reduced fat" ice creams are usually in the range of 5–7.5% fat. Lower fat versions are usually in the range of 3–5% fat. It has generally been possible to produce products as low as 4% fat, with traditional ingredients, but further fat reductions have

**Table 4** Suggested Mixes for Soft-Frozen Ice Cream Products (%)

Milk fat	10.0	10.0	
Milk solids-not-fat	12.6	12.0	
Sucrose	13.0	10.0	
Corn syrup solids	_	4.0	
Stabilizer <sup>a</sup>	0.15	0.15	
Emulsifier <sup>a</sup>	0.20	0.20	
Total solids	36.0	36.3	

<sup>&</sup>lt;sup>a</sup>Highly variable depending on type; manufacturers' recommendations are usually followed.

**Table 5** Suggested Mixes for Low-Fat Ice Cream or Ice Milk Products (3–5% Fat) and Light Ice Cream Products (6–8% fat)

Milk fat	3.0	4.0	5.0	6.0	8.0
Milk solids-not-fat	13.0	12.5	12.5	12.0	11.5
Sucrose	11.0	11.0	11.0	13.0	12.0
Corn syrup solids	6.0	5.5	5.5	4.0	4.0
Stabilizer <sup>a</sup>	0.35	0.35	0.35	0.35	0.35
Emulsifier <sup>a</sup>	0.10	0.10	0.10	0.15	0.15
Total solids	33.65	33.45	34.45	35.5	36.0

<sup>&</sup>lt;sup>a</sup>Highly variable depending on type; manufacturers' recommendations are usually followed.

generally involve the incorporation of fat-replacers. These are discussed further in Sec. B.1. Suggested formulations for light and low-fat ice creams are presented in Table 5.

#### 3. Sherbet

Sherbet is usually taken to be a frozen dairy dessert made from a milk product but containing a low (usually a legally defined maximum, e.g., 5%) level of milk solids, including milk fat, a high level of sweeteners (sugar and corn syrup solids, 30–35%), and added acidity (usually to greater than a legally defined minimum, e.g., 0.35%, expressed as lactic acid). Suggested formulations are given in Table 6. Because of the acidified nature of sherbets, they are most suited for typical acidic fruit flavors, e.g., citrus. The sugar and acid levels in fruits or fruit purees have to be considered in the final formulation and are included in the numbers suggested above. Acidity is usually added in the form of citric or tartaric acid, and this level of acidity modifies the perception of sweetness that would otherwise be created by the high level of sugars. Acid should not be added to ice and sherbet mixes until just before freezing. Heating some stabilizers in the presence of acid will reduce their effectiveness. Adding acid to a sherbet mix in which the milk solids have been included may result in aggregation/precipitation of the protein. Sherbet generally requires the addition of milk solids, and at least some fat ( $\sim 0.5\%$ ) is desirable as it tends to lubricate the dynamic freezer and provides a slightly more pleasant mouth feel than nonfat products. In many multiproduct manufacturing settings, ice cream mix is widely used as a source of milk solids, and the amount added will depend upon the level of milk solids desired. Overrun should be kept much lower in sherbet than that in ice cream, usually 30-35%.

**Table 6** Suggested Sherbet Mixes Showing Typical Components (%)

Milk fat	0.5	1.5
Milk solids-not-fat	2.0	3.5
Sucrose	24.0	24.0
Corn syrup solids	9.0	6.0
Stabilizer/emulsifier	0.3	0.3
Citric acid (50% sol.)	0.7	0.7
Water	63.5	64.0
Total	100.0	100.0

# 4. Frozen Yogurt

Yogurt is a well-established dairy product and is generally perceived to be characterized by developed acidity (lactic acid) from fermentation of lactose by bacterial culture and may or may not include live culture. The acidity destabilizes the casein micelles in the milk, and they in turn establish the typical acid gel. Frozen yogurt therefore should be much like the unfrozen version and be characterized also by developed acidity from fermentation. The example formulation in Table 7 is typical of a more traditional frozen yogurt. However, in most legal jurisdictions, frozen yogurt is not standardized, so a wide range of products exists, including those in which the acidity is not developed by bacterial culture but has been added in the form of citric acid.

To make a traditional frozen yogurt, as in Table 7, the processing occurs in two steps, the manufacture of a fermented yogurtlike ingredient, and the blending of this product with the rest of the ingredients. For example, 20% of the mix in Table 7, consisting of skim milk and skim milk powder, blended to give 12.5% msnf, is pasteurized at 85–90°C, cooled to 40 to 43°C, inoculated with a yogurt culture (typical of yogurt processing), and incubated as the yogurt portion. When the fermentation is complete (to the desired acidity), the "yogurt" is cooled. To make the "sweet" mix, the cream, sugar, stabilizer, and the balance of the skim milk powder and skim milk are combined, pasteurized, homogenized, cooled (typical for ice cream processing), and then blended with the "yogurt." The completed frozen yogurt mix is then aged and prepared for flavoring and freezing.

#### 5. Fruit Ices and Sorbets

"Ice" or "sorbet" is likewise typically not defined in legal regulations but is generally taken to be much the same as sherbet except that milk solids are not included. Sorbets are generally frozen in a swept surface freezer, while ices are generally frozen quiescently in molds. Both sorbets and ices are usually fruit based, and ingredients include combinations of fruit and/or fruit juices, sugar, stabilizer, and water. Overrun is very low, as aeration is difficult to achieve without protein or emulsifier. To make water ice or sorbet mixes from the above-suggested sherbet formulae, delete the fat and msnf.

# B. Sources and Functional Roles of Ingredients

#### 1. Fat

The fat component of frozen dairy dessert mixes increases the richness of flavor, produces a characteristic smooth texture by lubricating the palate, helps to give body, and aids in producing desirable melting properties (1,6). The fat content of a mix also aids in

**Table 7** Suggested Frozen Yogurt Formulation (%)

Milk fat	2.0
Milk solids-not-fat	14.0
Sugar	15.0
Stabilizer	0.35
Water	68.65
Total	100.0

lubricating the freezer barrel while the ice cream is being manufactured. Limitations on excessive use of fat in a mix include cost, a hindered whipping ability, decreased consumption due to excessive richness, and high caloric value. Fat contributes 9 kCal/g to the diet, regardless of its source. During freezing of ice cream, the fat emulsion that exists in the mix will partially coalesce (destabilize) or churn as a result of emulsifier action, air incorporation, ice crystallization, and high shear forces of the blades (6, 12).

This partial churning is necessary to set up the structure and texture in ice cream, which is very similar to the structure in whipped cream (13). This process will be discussed in Sec. II.B.4. The fat content is an indicator of the perceived quality and/or value of the ice cream. Ice cream must have a minimum fat content of 10% in most legal jurisdictions. Premium ice creams generally have fat contents of 14 to 18%. It has become desirable, however, to create light ice creams, < 10% fat, with the same perceived quality. In addition to structure formation, fat contributes a considerable amount of flavor to ice cream, which is difficult to reproduce in low-fat ice creams. Fat content must be altered by at least 1% before any noticeable difference appears in the taste or texture (1). Several recent papers have examined the effect of source and quantity of milk fat on sensory and textural characteristics of ice cream (14–20).

Milk fat as a fat source for ice cream formulations is in widespread use in North America, Australia, and New Zealand and parts of Europe. The triglycerides in milk fat have a wide melting range,  $+40^{\circ}$  to  $-40^{\circ}$ C. The crystallization patterns of milk fat are also very complex, owing in part to the large variation in fatty acids and large numbers of different triglycerides present (21). Consequently, there is always a combination of liquid and crystalline fat at refrigeration and subzero temperatures. Alteration of this solid: liquid ratio at freezer barrel temperatures, through natural variation or fat fractionation, may affect the ice cream structure formed. The best source of milk fat in ice cream for high-quality flavor is fresh sweet cream, from fresh sweet milk (1). Other sources of milk fat include sweet (unsalted) butter, frozen cream, or condensed milk blends. Whey creams have also been used but may lead to flavor or texture problems.

Vegetable fats are used extensively as fat sources in ice cream in the United Kingdom and parts of Europe, but only to a very limited extent in North America. Three factors of great interest in the selection of the fat source are the way in which the fat crystallizes, the temperature-dependent melting profile of the fat, especially at refrigerator and freezer temperatures, and the flavor and purity of the oil (6). For optimal partial coalescence during freezing, it is important that the fat droplet contain an intermediate ratio of liquid: solid fat at the time of freezing. Crystallization of fats occurs in three steps: subcooling of the oil (below the equilibrium crystallization temperature) to induce nucleation, heterogeneous or homogeneous nucleation (or both), and crystal propagation. In bulk fat, nucleation is predominantly heterogeneous, with crystals themselves acting as nucleating agents for further crystallization, and subcooling is usually minimal. However, in an emulsion, each droplet must crystallize independently of the next. For heterogeneous nucleation to predominate, there must be a nucleating agent available in every droplet, which is often not the case. Thus in emulsions, homogeneous nucleation and extensive subcooling are expected (6). Blends of oils are often used in ice cream manufacture, selected to take into account physical characteristics, flavor, availability, and cost. Hydrogenation is often necessary to achieve the appropriate melting characteristics. Palm kernel oil, coconut oil, palm oil, sunflower oil, peanut oil, and fractions and thereof, with varying degrees of hydrogenation, are all used to some extent. Tong and coworkers (22) substituted a portion of milk fat in ice cream with safflower oil, a highly unsaturated oil, in an attempt to lower the saturated fatty acid content of the final product. They reported

that increasing concentration of safflower oil decreased overrun but had little effect on the extent of fat destabilization at lower substitution levels.

There has been a great interest in the marketplace in the development of lower fat alternatives to traditional ice cream products. As a result, a large amount of product development time has been used in searching for a combination of ingredients that will replace the textural and flavor characteristics of fat in ice cream (17, 18, 23). These often involve the use of fat substitutes. Such products may be formulated with starch or other polysaccharides, proteins, or lipids, but their main requirement is to provide fewer calories to the product than traditional fat sources in the diet. A great deal of technical literature exists on the various properties of the products being marketed by a number of commercial firms. Schmidt and coworkers (24) studied the rheological, freezing, and melting properties of ice milks manufactured with protein-based or maltodextrin-based fat alternatives. They concluded that the carbohydrate-based alternatives resulted in greater effects on mix rheology while the protein-based alternatives were more similar to ice cream, owing in part to the functional contributions of proteins to food systems, especially in the area of emulsification and air incorporation. Ice cream products are very complex systems, both in structure and in flavor. In creating products that are meant to deliver to the consumer the same attributes but with less fat or fewer calories, it is imperative that the structural element of fat be considered to the same extent as flavor in order to deliver highquality products and develop market share for these products.

#### 2. Milk Solids-Not-fat

Milk solids-not-fat (msnf) or serum solids improve the texture of ice cream, aid in giving body and chew resistance to the finished product, are capable of allowing a higher overrun without the characteristic snowy or flaky textures associated with high overruns, and may be a cheap source of total solids (25). The msnf contain the lactose, caseins, whey proteins, minerals (ash), vitamins, acids, enzymes, and gases of the milk or milk products from which they were derived. The content of msnf used in a mix can vary from 10 to 14% or more. Whole milk protein blends contain both caseins and whey proteins, and this category includes most of the traditional sources of milk msnf, fresh concentrated skimmed milk, or spray-dried low-heat skim milk powder. However, most ice cream formulations now use another source or sources of msnf or milk protein to replace all or a portion of skim milk solids, for both functional and economical reasons (26).

When assessing replacements for skim milk solids, an important consideration is the levels of protein, lactose, and ash in the ingredients being assessed (27). Lactose is not very sweet and not very soluble, and therefore during the freezing of ice cream, it is freeze-concentrated beyond maximum solubility (supersaturated) and thus potentially prone to crystallization. Lactose crystals are very undesirable in ice cream, causing the defect known as sandiness. Lactose, being a disaccharide, also contributes to freezing point depression in the mix, so its concentration must be controlled closely. In addition, the milk salts affect both the flavor and the texture of ice cream. Also, when replacing skim milk solids, sufficient total solids must be added to limit the water content of the mix and meet legal minimum total solids requirements. For these reasons, it is often desirable to replace skim milk solids with a product or products with similar concentrations of lactose and protein. Lactose can be reduced through ultrafiltration or modified by limited hydrolysis to its constituent monosaccharides; either change will affect the concentration of the ingredient that can be used and the subsequent protein level achieved in the ice cream. Buttermilk solids have often been cited as a useful substitute for skim milk solids.

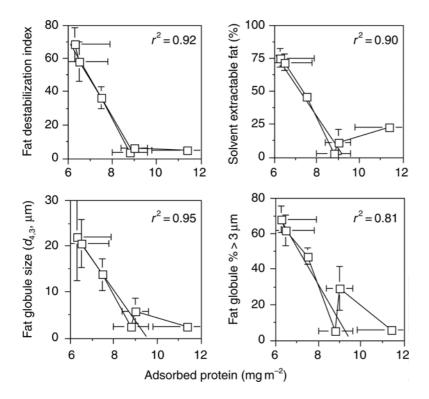
Buttermilk contains a higher concentration of fat globule membrane phospholipids than skim milk. Thus it can be used for its emulsifying properties to reduce the need for emulsifiers, or in formulations where it is undesirable to add emulsifiers (1).

It is possible to produce concentrated protein products from the casein portion of milk proteins, the most common for use as a food ingredient being sodium caseinate. The use of sodium caseinate in ice cream has been investigated, and a small percentage may be useful in contributing to functional properties, particularly aeration and emulsification (28, 29). However, the functionality of sodium caseinate is different from that of micellar casein, the form in which it is found in milk ingredients, and this needs to be considered when proposing its use. It can contribute positively to aeration, but it may lead to an emulsion that is too stable to undergo the required degree of partial coalescence. It is therefore most desirable in the serum phase rather than at the fat interface.

There has been a great deal of attention to the use of whey products in ice cream. Whey contains fat, lactose, whey proteins and water but very little, if any, casein. While skim milk powder contains 54.5% lactose and 36% protein, whey powder contains 72–73% lactose and only about 10–12% protein. Thus it can aggravate some of the problems associated with high lactose. However, an increasing number of whey products are available that have higher protein and lower lactose contents, mostly processed by membrane technology. Many of these can provide much higher quality than the traditional whey ingredients (26, 29). Whey protein concentrates with similar protein and lactose contents to skim milk solids can be produced. Protein content can vary from low values of 20–25% to 75% or more. In addition, the level of lactose can be modified by hydrolysis, although the freezing point depression effect of the higher monosaccharide content must be considered. Ash content can be reduced by demineralization. Whey protein isolates, which contain no lactose, are also available for blending with other ingredients to form the msnf content of ice cream formulations.

Proteins contribute much to the development of structure in ice cream, including emulsification, whipping, and water holding capacity (8, 30). The interfacial behavior of milk protein in emulsions is well documented, as is the competitive displacement of proteins by small molecule surfactants (31-35). In ice cream, the emulsion must be stable to withstand mechanical action in the mix state but must undergo sufficient partial coalescence to establish desirable structural attributes when frozen. These include dryness at extrusion for fancy molding, slowness of melting, some degree of shape retention during melting, and smoothness during consumption. This implies the use of small molecule surfactants (emulsifiers) to reduce protein adsorption and produce a weak fat membrane that is sensitive to shear action (7, 11, 12, 29, 36–41). Bolliger and coworkers (42) showed that protein adsorbed to the fat droplets (mg m<sup>-2</sup>) in ice cream mix correlated with major characteristic analyses describing the fat structure in ice cream (fat agglomerate size, fat agglomeration index, solvent extractable fat) (Fig. 1). The loss of steric stability from the globule, which was contributed from micellar adsorption, accounts for its greater propensity for partial coalescence during shear. Partial coalescence is responsible for establishing a three-dimensional aggregation of fat globules that provide structural integrity (see Sec. II.B.4). This is especially important if such integrity is needed when the structural contribution from ice is weaker (i.e., before hardening or during melting). Variables that affect the destabilization of fat in ice cream have been well studied (43–46).

With respect to protein contribution to fat globule integrity, it is obvious from the studies to date that a weak surface layer is most desirable (8). Segall and Goff (47) examined the susceptibility of ice cream emulsions to partial coalescence during shear



**Figure 1** The effect of protein adsorbed to the fat globules in the mix on fat destabilization index, solvent extractable fat, and fat agglomerate size in ice cream. (From Ref. 42.)

when the emulsion was prepared with varying concentrations and types of protein while still retaining sufficient quiescent emulsion stability. The membranes of fat globules stabilized by whey protein isolate were more susceptible than those made from sodium caseinate or casein micelles, while those made from partially hydrolyzed whey proteins did not show sufficient quiescent emulsion stability. However, when casein was added to the whey protein—stabilized emulsion, after homogenization, further casein adsorption to the whey protein membrane was rapid. Nevertheless, an understanding of protein structures and protein: surfactant interactions at the fat interface may lead to better control over the extent of partial coalescence desirable in the finished product.

Milk proteins are well known for their foaming properties, and during the manufacture of ice cream, air is incorporated to about 50% phase volume. Thus it should not be surprising that milk proteins contribute to stabilizing the air interface in ice cream. This air interface is very important for overall structure and structural stability (48). Loss of air can lead to a defect known as shrinkage (see Sec. III.B.), the occurrence of which is fairly common and very significant for quality loss and unacceptability of the product (49). The process of whipping heavy cream includes an initial protein adsorption to the air interface and a subsequent adsorption of fat globules and their associated membrane to the existing protein air bubble membrane (13). Globular fat adsorption to air interfaces is known to stabilize air bubbles from rapid collapse (50). Proteins at the fat interface have also been shown to play an important role during the aeration of emulsions (51).

However, the actual contribution of protein to ice cream aeration, and its interaction with both the added emulsifying agents (which are also surface active) and

partially coalescing fat at the air interface, has been less well studied. Incorporation of air into ice cream is rapid, within seconds, and at the same time the viscosity of the surrounding matrix is increasing exponentially owing to freezing, so that air bubbles after formation become physically trapped in a semisolid matrix, making their collapse quite difficult.

As with the role of milk protein in aeration, its role in the unfrozen aqueous phase is recognized but less well studied than its role at the fat interface. Milk proteins interact with water, and the subsequent hydration is responsible for a variety of functional properties, including rheological behavior. Thus freeze-concentration of proteins in ice cream must lead to a sufficient concentration to have a large impact on the viscosity of the unfrozen phase and its subsequent effect on ice crystallization, ice crystal stability, and solute mobility (52). Jonkman and coworkers (53) studied the effect of ice cream manufacture on the structure of casein micelles and found that the micelles per se were not affected by the process. Although the stability of the micelle was expected to be affected by low temperature, this was offset by an increasing concentration of milk salts in solution during freeze-concentration, so that the micelle remained intact in a similar state to that found in mix.

Polysaccharides are also added to ice cream mix to enhance solution viscosity and to impact on ice crystallization behavior. Commonly used polysaccharides can be incompatible in solution with milk proteins, leading to a microscopic or macroscopic phase separation (54), a phenomenon that has been studied in milk and ice cream—type systems (55–57). Goff and coworkers (58) examined the interaction between milk proteins and polysaccharides in frozen systems using labeled polysaccharides and fluorescence microscopy and demonstrated a clear phase separation between the two, leading to discernable networks created by freezing from both locust bean gum and milk proteins. It has also been shown in ice cream that when in solution with polysaccharides, the casein aggregates into distinct networks (58). Flores and Goff (59) demonstrated that milk proteins had a large impact on ice crystal size and stability. It thus appears that microscopic phase separation of the milk protein induced by polysaccharides, and "aggregation" of casein into a weak gellike network, promoted also by freeze-concentration, may be at least partly responsible for ice crystal stability and thus the improvement of texture during consumption.

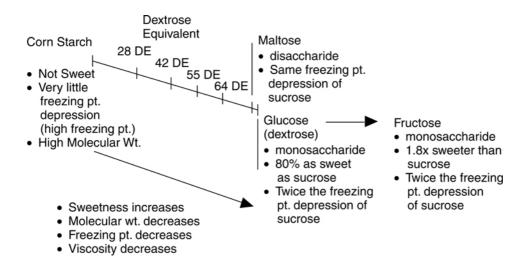
Lactose, or milk sugar, is a disaccharide of glucose and galactose that does not contribute much to sweetness of ice cream, since it is only 1/5 to 1/6 as sweet as sucrose (21). Lactose is relatively insoluble and crystallizes in two main forms, an  $\alpha$ monohydrate and a  $\beta$  anhydrous, depending on conditions. The  $\alpha$  monohydrate crystals, which take on a characteristic tomahawk shape, lead to the defect known as sandiness when they are allowed to grow sufficiently large (about 15 µm). Lactose content of ice cream mix is about 6% if no whey powder has been used in the formulation. Levels of lactose in ice cream mix in excess of this leads to a reduced freezing point, causing a softening of the ice cream and the potential for development of iciness, a greater potential for lactose crystallization, or sandiness, and salty flavors (60). The lactose solubility in water at room temperature is about 11% (21). During freezing, this concentration is exceeded as a result of freeze concentration (water removal in the form of ice). When 75% of the water is frozen in a mix consisting originally of 11% msnf (6% lactose), the lactose content in the unfrozen water corresponds to  $\sim 40\%$ . Probably much of the lactose in ice cream exists in a supersaturated, amorphous (noncrystalline) state, but owing to extreme viscosity it does not crystallize (61). Stabilizers help to hold lactose in a supersaturated state due to viscosity enhancement.

#### 3. Sweeteners

Sweet ice cream is usually desired by the consumer. As a result, sweetening agents are added to ice cream mix at a rate of usually 12–17% by weight. Sweeteners improve the texture and palatability of ice cream, enhance flavors, and are usually the most economical source of total solids (1). Their ability to lower the freezing point of a solution imparts a measure of control over the temperature–hardness relationship (see Sec. II.B.1). In determining the proper blend of sweeteners for an ice cream mix, the total solids required from the sweeteners, the sweetness factor of each sugar, and the combined freezing point depression of all sugars in solution must be calculated to achieve the proper solids content, the appropriate sweetness level, and a satisfactory degree of hardness (5, 6, 62). The most common sweetening agent used is sucrose, alone or in combination with other sugars. Sucrose, like lactose, is most commonly present in ice cream in the supersaturated or glassy state, so that no sucrose crystals are present (6, 61).

It has become common practice in the industry to substitute sweeteners derived from corn starch or other starch sources such as rice for all or a portion of the sucrose (1, 4). A typical sweetener blend for an ice cream mix usually includes 10–12% sucrose and 4–5% corn syrup solids (corn starch hydrolysate syrup, commonly referred to as "glucose solids") (1, 4). The use of corn syrup solids in ice cream is generally perceived to provide enhanced smoothness by contributing to a firmer and more chewy texture, to provide better meltdown characteristics, to bring out and accentuate fruit flavors, to reduce heat shock potential, which improves the shelf life of the finished product, and to provide an economical source of solids (62, 63).

During the hydrolysis process, starch, a high-molecular-weight polymer of the monosaccharide glucose (dextrose), is continually and systematically cleaved by enzymes ( $\alpha$  amylase, glucoamylase and  $\beta$  amylase) to produce mixtures of medium and low molecular weight units (Fig. 2). The medium molecular weight saccharides (dextrins) are effective stabilizers and provide maximum prevention against coarse ice crystal formation, which is reflected in improved meltdown and heat shock resistance. They also improve cohesive and adhesive textural properties. The smaller molecular weight sugars provide smoothness, sweetness, and flavor enhancement. With the appropriate use of enzyme



**Figure 2** An illustration of the products that result from the hydrolysis of corn starch and their properties relevant to ice cream manufacture.

technology, corn syrup manufacturers have the ability to control the ratios of high- to lowmolecular-weight components, and the ratios of maltose, the disaccharide, to glucose, the monosaccharide. Glucose monosaccharide offers sweetness synergism with sucrose, but, being half the molecular weight, has greater freezing point depression than either sucrose or maltose. The ratio of higher to lower molecular weight fractions can be estimated from the dextrose equivalent (DE) of the syrup. Figure 2 shows that as the DE decreases, the sweetness increases, but the freezing point decreases (more freezing point depression), and the contribution to viscosity and chewiness in the mouth decreases. Thus optimization of DE and concentration of corn sweeteners are required for the most beneficial effects. Ice cream manufacturers usually use a 28 to 42DE syrup, either liquid or dry (1,62). High maltose syrups modify the effect of dextrose on freezing point (62, 63). With further enzyme processing (glucose isomerase), glucose can be converted to fructose (Fig. 2), as in the production of high-fructose corn sweeteners. The resultant syrup is much sweeter than sucrose, although it has half the molecular weight and thus contributes more to freezing point depression than sucrose. These modifications to properties would also require optimization of all sugars for appropriate use of HFCS, although it has been shown that blends of high fructose syrup, high maltose syrup, and low DE syrup can be utilized to provide appropriate sweetness, freezing point depression, and total solids, in the absence of sucrose (62, 63).

#### 4. Stabilizers

Ice cream stabilizers are a group of ingredients (usually polysaccharides) commonly used in ice cream formulations. The primary purposes for using stabilizers in ice cream are to produce smoothness in body and texture, to retard or reduce ice and lactose crystal growth during storage, especially during periods of temperature fluctuation, known as heat shock (64), and to provide uniformity to the product and resistance to melting (1,4). They also increase mix viscosity, stabilize the mix to prevent wheying off (e.g., carrageenan), aid in suspension of flavoring particles, produce a stable foam with easy cutoff and stiffness at the barrel freezer for packaging, slow down moisture migration from the product to the package or the air, and help to prevent shrinkage of the product volume during storage (65). Stabilizers must also have a clean, neutral flavor, not bind to other ice cream flavors, contribute to acceptable meltdown of the ice cream, and provide desirable texture upon consumption (65). Limitations on their use include production of undesirable melting characteristics, excessive mix viscosity, and contribution to a heavy, soggy body. Although stabilizers increase mix viscosity, they have little or no impact on freezing point depression.

Gelatin, a protein of animal origin, was used almost exclusively in the ice cream industry as a stabilizer but has gradually been replaced by polysaccharides of plant origin owing to their increased effectiveness and reduced cost (1). Stabilizers currently in use include: (a) carboxymethyl cellulose, derived from the bulky components or soluble fiber of plant material; (b) locust bean gum (carob bean gum), which is derived from the beans of the tree *Ceratonia siliqua*, grown mostly in the Mediterranean; (c) guar gum, from the guar bush, *Cyamoposis tetragonolba*, a member of the legume family grown in India and Pakistan for centuries and now grown to a limited extent also in the United States; (d) xanthan, a bacterial exopolysaccharide produced by the growth of *Xanthomonas campestris* in culture; (e) sodium alginate, an extract of another seaweed, kelp, a brown algae; or (f) carrageenan, an extract of *Chondus crispis* (Irish moss), a red algae, originally harvested from the coast of Ireland, near the village of Carragheen. Each stabilizer has its

own characteristics, and often two or more of these stabilizers are used in combination to lend synergistic properties to each other and improve their overall effectiveness. Guar, for example, is more soluble than locust bean gum at cold temperatures, thus it finds more application in HTST pasteurization systems. Carrageenan is a secondary colloid used to prevent wheying off of mix, which is usually promoted by one of the other stabilizers (1, 6), hence it is included in most blended stabilizer formulations.

The mechanisms by which ice cream stabilizers affect freezing properties or limit recrystallization (see Sec. III.B.1) have been extensively studied but are as yet not fully understood. Ice recrystallization in ice cream has recently been reviewed (10,66). It appears from the literature available to date that stabilizers have little (67) or no (68,69) impact on the initial ice crystal size distribution in ice cream at the time of the draw from the scraped surface heat exchanger, and also little or no impact on initial ice growth during quiescent freezing and hardening (52, 70, 71), but do limit the rate of growth of ice crystals during recrystallization (59, 67–69, 72–78). They have no effect on the freezing properties of an ice cream mix, e.g., freezing point depression (79, 80), amount of freezable water or enthalpy of melting (71, 81, 82), or heterogeneous nucleation (83), and thus may not have been expected to affect initial ice crystallization processes. With respect to recrystallization, there has not been a demonstrable correlation between the viscosity of the unfrozen mix and the recrystallization rate (74, 79, 80, 84). Their protective effect also appears not to be related to a modification of the glass transition (74, 82, 84, 85). However, it has been suggested that they modify the ice crystal serum interface, either through surface adsorption to the crystal itself (68, 69, 76, 78), or by modifying the rate at which water can diffuse to the surface of a growing crystal during temperature fluctuation, or the rate at which solutes and macromolecules can diffuse away from the surface of a growing ice crystal (67, 85), or by some other modification of the ice-serum interface (86). It must be remembered that freeze-concentration of the unfrozen phase results in a polysaccharide concentration several times higher than what was present in the original mix. Most polysaccharides are also incompatible in solution with milk proteins, which leads to further localized concentrations. Recent research by Goff and co-workers (58) has focused on the ability of at least some stabilizers to form a cryogel and entrap ice crystals within the gel. Phase separation of polysaccharides and proteins appears also to be related. Control of ice recrystallization may then relate to microstructural differences in solute concentration at the surface of the crystal.

#### 5. Emulsifiers

Emulsifiers have been used in ice cream mix manufacture for many years (87, 88). They are usually integrated with the stabilizers in proprietary blends, but their function and action is very different from the stabilizers. They are used for improvement of the whipping quality of the mix, for production of a drier ice cream to facilitate molding, fancy extrusion, and sandwich manufacture, for smoother body and texture in the finished product, for superior drawing qualities at the freezer to produce a product with good stand-up properties and melt resistance, and for more exact control of the product during freezing and packaging operations (1,87–89). Their mechanism of action can be summarized as follows: they lower the fat/water interfacial tension in the mix, resulting in protein displacement from the fat globule surface, which in turn reduces the stability of the fat globule to partial coalescence that occurs during the whipping and freezing process, leading to the formation of a fat structure in the frozen product that contributes greatly to texture and meltdown properties (12). The extent of protein displacement from the

membrane, and hence the extent of dryness achieved, is a function of the emulsifier concentration (6, 90). Their role in structure formation will be described further in Sec. II.B.

Egg yolk was formerly commonly used as an ice cream emulsifier. Emulsifiers used in ice cream manufacture today are of two main types: the mono- and diglycerides and the sorbitan esters. Mono- and diglycerides are derived from the partial hydrolysis of fats of animal or vegetable origin. The sorbitan esters are similar to the monoglycerides in that the sorbitan esters have a fatty acid molecule such as stearate or oleate attached to a sorbitol molecule, whereas the monoglycerides have a fatty acid molecule attached to a glycerol molecule. To make the sorbitan esters water soluble, polyoxyethylene groups are attached to the sorbitol molecule. Polysorbate 80, polyoxyethylene sorbitan monooleate, is the most common of these sorbitan esters. Polysorbate 80 is a very active drying agent in ice cream (12) and is used in many commercial stabilizer/emulsifier blends.

# II. MANUFACTURING AND STRUCTURE OF FROZEN DESSERT PRODUCTS

#### A. Mix Manufacture

Ice cream processing operations can be divided into two distinct stages, mix manufacture and freezing operations (Fig. 3). Ice cream mix manufacture consists of the following unit operations: combination and blending of ingredients, batch or continuous pasteurization, homogenization, and mix aging.

# 1. Blending

Ingredients are usually preblended prior to pasteurization, regardless of the type of pasteurization system used. Blending of ingredients is relatively simple if all ingredients are in the liquid form, as automated metering pumps or tanks on load cells can be used for measurement, and tanks, usually conical-bottom and agitated, are used for mixing. When dry ingredients are used, powders are added through either a pumping system under high velocity or through a liquifier, a large centrifugal pump with rotating knife blades that chop all ingredients as they are mixed with the liquid (3).

# 2. Mix Calculations

The general object in calculating ice cream mixes is to turn the formula, which is based on the desired components, into a recipe, which is based on the actual ingredients to be used to supply the components and the amount of mix desired. The formula is given as percentages of fat, msnf, sugar, corn syrup solids, stabilizers, and emulsifiers. The ingredients to supply these components are chosen on the basis of availability, quality, and cost. Table 8 illustrates the relationship between the major components, the main ingredients that supply the major components, and the minor components that are supplied with the major ones for each ingredient.

The first step in a mix calculation is to identify the composition of each ingredient. In some cases the percentage of solids contained in a product is taken as constant or provided by an ingredient supplier, while in others the composition must be obtained by analysis (e.g., the fat content in milk or cream). If there is only one source of the component needed for the formula, for example the stabilizer or the sugar, it is determined directly by multiplying the percentage needed by the amount needed, e.g., 100 kg of mix @ 10% sugar

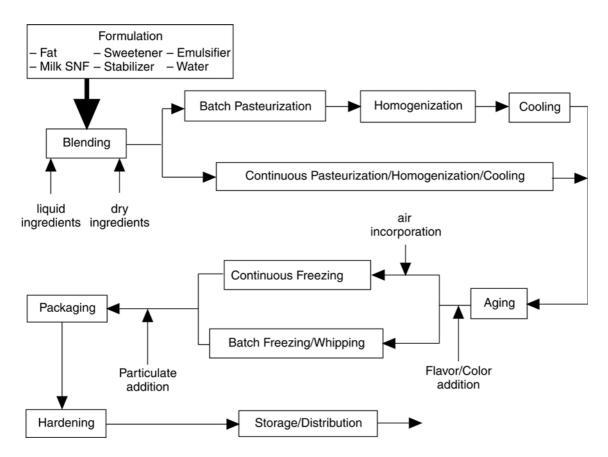


Figure 3 A schematic illustration of the processing steps in ice cream manufacture.

**Table 8** Sources of the Major Components in Ice Cream Mix, and the Minor Components Also Supplied by These Ingredients

Component	Ingredients to supply (but also supplies)	
Milk fat	Cream (msnf, water)	
	Butter (msnf, water)	
Milk solids-not-fat (msnf)	Skim powder (water)	
` ,	Condensed skim (water)	
	Condensed milk (water, fat)	
	Sweetened condensed (water, sugar)	
	Whey powder (water)	
Water	Skim milk (msnf)	
	Milk (fat, msnf)	
	Water	
Sweetener	Sucrose	
	Corn syrup solids	
	Liquid sugars (water)	
Stabilizers/emulsifiers	,	

would require  $10 \, \text{kg}$  sugar. If there are two or more sources, for example 10% fat coming from both cream and milk, then an algebraic method may be utilized. Computer programs developed for mix calculations generally solve simultaneous equations based on mass and component balances. For manual calculations, a method known as the serum point method has been derived (1,4). This method has solved the simultaneous equations in a general way so that only the equations need to be known and not resolved each time. In the serum point method, 9% msnf is assumed in the aqueous (serum), nonfat portion of all milk ingredients. Thus the msnf content of milk or cream is calculated as  $(100 - \text{percent fat}) \times 0.09$ . This section will illustrate mix calculation solutions using algebraic techniques and the serum point method.

# **Example Problem 1**

Mix using cream, skim milk, and skim powder (three sources of msnf, three sources of water). Solution shown by both the algebraic and the serum point methods.

Desired: 100 kg mix @ 13% fat, 11% msnf, 15% sucrose, 0.5% stabilizer, 0.15% emulsifier.

On hand: Cream @ 40% fat; skim milk; skim milk powder @ 97% msnf; sugar; stabilizer; emulsifier.

Solution via an Algebraic Method. Note: Only one source of fat, sugar, stabilizer, and emulsifier, but two sources of msnf.

Cream 100 kg mix 
$$\times \frac{13 \text{ kg fat}}{100 \text{ kg mix}} \times \frac{100 \text{ kg cream}}{40 \text{ kg fat}} = 32.5 \text{ kg cream}$$

Sucrose 100 kg mix  $\times \frac{15 \text{ kg sucrose}}{100 \text{ kg mix}} = 15 \text{ kg sucrose}$ 

Stabilizer 100 kg mix  $\times \frac{0.5 \text{ kg stabilizer}}{100 \text{ kg mix}} = 0.5 \text{ kg stabilizer}$ 

Emulsifier 100 kg mix  $\times \frac{0.15 \text{ kg emulsifier}}{100 \text{ kg mix}} = 0.15 \text{ kg emulsifier}$ 

Skim milk and skim powder. Note: Two sources of the msnf.

Now, let x = skim powder, y = skim milk.

MASS BALANCE. (All the components add up to  $100 \,\mathrm{kg}$ , so skim powder + skim milk =  $100 - \mathrm{mass}$  of other ingredients.)

$$x + y = 100 - (32.5 + 15 + 0.5 + 0.15)$$

MSNF BALANCE. Equal to 11% of the mix and coming from the skim milk, the skim powder, and the cream, so the portion from the skim powder and skim milk = 11 kg - the contribution from the cream. The msnf portion of the skim milk and cream are taken as 9% of the nonfat portion, i.e., 9% in the case of the skim milk and  $(100-40) \times 0.09 = 5.4\%$  in the case of the cream.

$$0.97x + 0.09y = 0.11(100) - (0.054 \times 32.5)$$

Once the appropriate equations have been written, they need to be solved algebraically.

$$x + y = 51.85$$
 so  $y = 51.85 - x$  From the mass balance

$$0.97x + 0.09y = 9.245$$
  
 $0.97x + 0.09(51.85 - x) = 9.245$   
 $0.97x - 0.09x + 4.67 = 9.245$   
 $0.88x = 4.58$   
 $x = 5.20 \text{ kg skim powder}$   
 $y = 46.65 \text{ kg skim milk}$ 

From the msnf balance Substituting

This shows the simultaneous solution of two equations with two unknowns. Likewise, if there were three unknowns, e.g., fat, msnf, and the total weight, then three equations could be written, one for each of fat, msnf, and weight. However, the above problem could also be solved with the serum point method, and the solution of the above example by that method, along with the derivation of the equations, follows. The serum point calculation assumes 9% msnf in skim milk and the skim portion of all dairy ingredients. It then solves the calculation beginning with the most concentrated source of msnf first. It is advisable to solve a problem with the serum point method on the basis of  $100 \, \text{kg}$ , and then scale it up to the desired mix quantity by multiplying by the appropriate factor, e.g., solution for each component for  $100 \, \text{kg} \times 50 = \text{solution}$  for  $5000 \, \text{kg}$ .

#### Solution of Problem 1 via the Serum Point Method

1. Amount of skim milk powder needed is found by the following formula:

$$\frac{\text{msnf needed} - (\text{serum of mix} \times 0.09)}{\text{% msnf in powder} - 9} \times 100 = \text{kg skim powder}$$
 (1)

The derivation of Eq. (1) is shown at the end of the problem. For now, just assume that this equation will work.

The serum of the mix is found by adding the desired percentages of fat, sucrose, stabilizer, and emulsifier together and subtracting from 100 [i.e., "serum" = msnf (or "serum solids") + water]. In the present problem then,

$$100 - (13 + 15 + 0.5 + 0.15) = 71.35 \,\mathrm{kg}$$
 serum

Substituting in Eq. (1) we have

$$\frac{11 - (71.35 \times 0.09)}{97 - 9} \times 100 = \frac{4.58}{88} \times 100 = 5.20 \,\text{kg skim powder}$$

2. The weight of cream (since there is only one source of fat) will be

$$13 \text{ kg} \times \frac{100 \text{ kg cream}}{40 \text{ kg fat}} = 32.5 \text{ kg cream}$$

- 3. The sucrose will be 15 kg/100 kg mix.
- 4. The stabilizer will be 0.5 kg/100 kg mix.
- 5. The emulsifier will be  $0.15 \,\mathrm{kg}/100 \,\mathrm{kg}$  mix.
- 6. The weight of mix supplied so far is

Cream	32.50 kg
Skim powder	$5.20\mathrm{kg}$
Sucrose	$15.00\mathrm{kg}$
Stabilizer	$0.50\mathrm{kg}$
Emulsifier	$0.15  \mathrm{kg}$
	53.35 kg

The skim milk needed therefore is  $100 - 53.35 = 46.65 \,\mathrm{kg}$ .

It is always important to check your solutions to ensure they give the desired result. Such a proof is shown here, where the quantities of each ingredient and the quantities of each component in each ingredient are laid out in a table and summed. The total mass of each component divided by the total mass of mix should yield the desired percentage.

#### **Proof**

Ingredients	Kilograms	kg fat	kg msnf	kg Total solids
Cream	32.50	13.0	1.75	14.75
Skim milk	46.65		4.20	4.20
Skim powder	5.20		5.05	5.05
Sucrose	15.00			15.00
Stabilizer	0.50		_	0.50
Emulsifier	0.15			0.15
Totals	100.0	13.0	11.0	39.65

Derivation of the serum point equations:

Problem 1 is resolved again using simultaneous equations in a general way to show where the serum point equations come from.

# **Solution**

Only one source of fat, sucrose, stabilizer, and emulsifier:

kg fat =  $100 \,\text{kg}$  mix  $\times$   $13 \,\text{kg}$  fat/ $100 \,\text{kg}$  mix =  $13 \,\text{kg}$  fat (The explanation for this assumption becomes clearer in a moment!)

kg sucrose =  $100 \text{ kg mix} \times 15 \text{ kg sucrose}/100 \text{ kg mix} = 15 \text{ kg sucrose}$ 

kg stabilizer =  $100 \text{ kg mix} \times 0.5 \text{ kg stab}/100 \text{ kg mix} = 0.5 \text{ kg stabilizer}$ kg emulsifier =  $100 \text{ kg mix} \times 0.15 \text{ kg emul}/100 \text{ kg mix} = 0.15 \text{ kg emulsifier}$ 

Two sources of msnf:

Let 
$$X = \text{skim powder (kg)}$$

Let Y = skim milk (kg) + skim milk in cream (kg)

MASS BALANCE

$$X + Y = \text{Total mix} - \text{components already added}$$

$$X + Y = 100 - (13 + 15 + 0.5 + 0.15)$$

(the "Serum of the Mix")

$$X + Y = 71.35$$

(so 
$$Y = 71.35 - X$$
)

MSNF BALANCE

$$0.97X + 0.09Y = (0.11 \times 100)$$

"Serum solids "Serum solids "Serum solids

fraction fraction fraction in powder" in skim" in mix"

$$0.97X + 0.09(71.35 - X) = 11$$

$$0.97X + (0.09 \times 71.35) - 0.09X = 11$$

$$0.97X - 0.09X = 11 - (0.09 \times 71.35)$$

$$X = \frac{11 - (0.09 \times 71.35)}{0.97 - 0.09}$$

Which is equal to

kg skim powder = 
$$\frac{\text{msnf needed} - (0.09 \times \text{serum of mix})}{\% \text{ msnf in powder} - 9} \times 100$$
 Which is Eq. (1)

$$X = \frac{4.58}{0.88} = 5.20 \,\mathrm{kg}$$
 powder

kg cream =  $13 \text{ kg fat} \times 100 \text{ kg cream}/40 \text{ kg fat} = 32.5 \text{ kg cream}$ 

kg skim = 
$$100 - 32.5 - 15 - 0.5 - 0.15 - 5.2$$
  
=  $46.65 \,\mathrm{kg}$ 

# **Example Problem 2**

Mix using cream, milk, and skim powder (three sources of msnf, three sources of water, and two source of fat). Solved by both the algebraic and the serum point methods.

Desired: 100 kg mix containing 14% fat, 9.5% msnf, 15% sucrose, 0.4% stabilizer,

1% frozen egg yolk.

On hand: Cream 30% fat, milk 3.5% fat, skim milk powder 97% solids, sucrose,

stabilizer, and egg yolk (50% solids).

The solution to this problem will be shown by the simultaneous solution of three equations, since there are three sources of msnf, three sources of water, and two sources of fat; and by the serum point method. Both produce the same results. Follow whichever method you prefer. Computer programs exist that solve simultaneous equations; writing the equations, however, requires an understanding of the objectives of the problem.

# Solution via the Algebraic Method

Sucrose: 
$$100 \text{ kg mix} \times \frac{15 \text{ kg sucrose}}{100 \text{ kg mix}} = 15 \text{ kg sucrose}$$

Stabilizer:  $100 \text{ kg mix} \times \frac{0.4 \text{ kg stabilizer}}{100 \text{ kg mix}} = 0.4 \text{ kg stabilizer}$ 

Egg yolk:  $100 \text{ kg mix} \times \frac{1 \text{ kg egg yolk}}{100 \text{ kg mix}} = 1 \text{ kg egg yolk}$ 

Now, let x = skim powder, y = milk, z = cream.

 $MASS\ BALANCE$ . All the components add up to  $100\,\mathrm{kg}$ , so the sum of the three unknowns = 100 – the sum of the known mass of the other components.

$$x + y + z = 100 - (15 + 0.4 + 1)$$

MSNF BALANCE. Equal to 9.5% of the mix and coming from the milk, the skim powder, and the cream; assume 9% in the skim portion of the milk and cream so that the msnf of the milk =  $0.09 \times (100 - 3.5)$  and of the cream =  $0.09 \times (100 - 30)$ 

$$0.97x + 0.08685y + 0.063z = 0.095(100)$$

FAT BALANCE. Equal to 18% of the mix and coming from the milk and cream 0.035y + 0.3z = 0.14(100)

These equations can now be solved to produce the final outcome:

$$x = 3.7 \text{ kg skim powder}$$
  
 $y = 37.7 \text{ kg milk}$   
 $z = 42.3 \text{ kg cream}$ 

#### Solution via the Serum Point Method

1. Determine the amount of skim milk powder required by using Eq. (1):

$$\frac{\text{msnf needed} - (\text{serum of mix} \times 0.09)}{\text{% msnf in powder} = 9} \times 100 = \text{skim powder}$$

Serum of mix = 100 - (14 + 15 + 0.4 + 1.0) = 69.6. Substituting, we have

$$\frac{9.5 - (69.6 \times 0.09)}{97 - 9} \times 100 = \frac{3.236 \times 100}{88} = 3.68 \,\text{kg powder}$$

- 2. Amount of sucrose required is 15.0 kg.
- 3. Amount of stabilizer required is 0.4 kg.
- 4. Amount of egg required is 1.0 kg.

- 5. Determine amount of milk and cream needed. Materials supplied so far are  $3.68 \, \text{kg}$  powder,  $15 \, \text{kg}$  sucrose,  $0.4 \, \text{kg}$  stabilizer, and  $1 \, \text{kg}$  egg yolk, a total of  $20.08 \, \text{kg}$ .  $100 20.08 = 79.92 \, \text{kg}$  milk and cream needed.
  - 6. Determine the amount of cream by the following formula:

$$\frac{\text{kg fat needed} - \left(\text{kg cream and milk needed} \times \frac{\% \text{ fat in milk}}{100}\right)}{\% \text{ fat in cream} - \% \text{ fat in milk}} \times 100$$
 (2)

*Note*: Eq. (2) is derived from a generalized fat balance, in much the same way that Eq. (1) was derived.

Substituting, we have

$$\frac{14 - (79.92 \times \frac{3.5}{100})}{30 - 3.5} \times 100 = \frac{11.20}{26.5} \times 100 = 42.26 \,\text{kg cream}.$$

7. Amount of milk needed = 79.92 - 42.26 = 37.66 kg of milk.

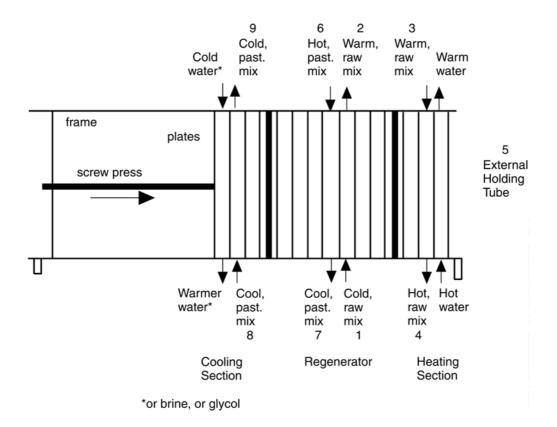
#### **Proof**

Ingredients	Kilograms	kg fat	kg msnf	kg Total solids
Cream	42.26	12.68	2.66	15.34
Milk	37.66	1.32	3.27	4.59
Skim powder	3.68		3.57	3.57
Sucrose	15.00	_	_	15.00
Stabilizer	0.40		_	0.40
Egg yolk	1.00		_	0.50
Totals	100.00	14.00	9.50	39.40

With Eqs. (1) and (2), most complex mix problems can be solved. There are additional complications for the use of condensed skim or whole milk and for liquid sugars. See Ref. (1) for further details.

# 3. Pasteurization and Food Safety Issues

Pasteurization is the biological control point in the system, designed for the destruction of pathogenic bacteria. If raw milk or cream are used as ingredients, it could be that these are contaminated with a human pathogen from the dairy farm. Therefore it is essential that pasteurization be carefully designed and closely monitored. If raw dairy ingredients are not used, contamination from a human source could also occur, and thus the use of pasteurization conditions that eliminate pathogens is mandated by most legal jurisdictions. In addition, it serves a useful role in reducing the total bacterial load, and in solubilization of some of the components (proteins and stabilizers). Both batch and continuous (high-temperature short-time or HTST) systems are in common use (3). In a batch pasteurization system, blending of the proper ingredient amounts is done in large jacketed vats equipped with some means of heating, usually saturated steam or hot water. The product is then heated in the vat to at least 69°C (155°F) and held for 30 min to satisfy legal requirements for pasteurization, or equivalent times and temperatures as determined by the local legal jurisdiction. The heat treatment must be severe enough to ensure destruction of pathogens and to reduce the bacterial count to a maximum (e.g.,



**Figure 4** A schematic illustration of the side view of a plate heat exchanger used for HTST pasteurization of frozen dairy dessert mixes. Numbers indicate the sequence of flow of the mix. Italics are used to differentiate the material on one side of a section from the material on the other.

10,000 per gram), depending on the legal jurisdiction. Following pasteurization, the mix is homogenized using high pressures and then is passed across some type of heat exchanger (plate heat exchanger or double or triple tube heat exchanger) for the purpose of cooling the mix to refrigerated temperatures (4°C).

Continuous pasteurization is usually performed in an HTST heat exchanger following the blending of ingredients in a large insulated feed tank. Some preheating, to 30 to 40°C, may be necessary for solubilization of the components. The HTST system is equipped with a heating section, a cooling section, and a regeneration section (Fig. 4). Mix first enters the raw regeneration section, where cold or preheated mix is heated to as warm as possible on one side of a plate heat exchanger while the pasteurized hot mix is cooled to as low as possible running countercurrent on the opposite sides of the plates. Following the raw regeneration section, mix enters the heating section where a minimum temperature of 80°C is obtained. Mix is held at this temperature for 25 s by flowing either through a series of holding tubes or through an additional set of plates in the HTST unit. Holding times much longer than the minimum can be accomplished with longer holding tubes. Holding times of 2 or 3 min may produce superior mixes that retain many of the advantages of batch pasteurization (4,6). After leaving the holding tube, mix enters the homogenizer, depending upon the particular configuration, then flows back through the pasteurized side of the regeneration section and enters the cooling plates where a chilled brine solution or chilled water bring the mix down to around 4°C.

# 4. Homogenization

Homogenization is responsible for the formation of the fat emulsion by forcing the hot mix through a small orifice under pressures of 15.5 to 18.9 MPa (2000 to 3000 psig), depending on the mix composition. The actual mechanism of fat disruption within the homogenizer is thought to result from turbulence, cavitation, and velocity gradients (energy density) within the valve body (91). The 4 to 8-fold increase in the surface area of the fat globules is responsible in part for the formation of the fat globule membrane, composed of adsorbing materials attempting to lower the interfacial free energy of the fat globules (92, 93). Koxholt and coworkers (94) have recently shown that sufficient fat structure in the mix for optimal ice cream meltdown was created by homogenization pressures on the first stage of 10 MPa, in mixes with up to 10% fat content, and that higher pressures were not required. With single-stage homogenizers, fat globules tend to cluster as bare fat surfaces come together, or adsorbed molecules are shared. Therefore a second homogenizing valve is commonly placed immediately after the first with applied back pressures of 3.4 MPa (500 psig) (3), allowing more time for surface adsorption to occur. However, Koxholt and coworkers (94) have recently shown that two-stage homogenization is not necessary for ice cream mixes up to 10% fat content, to achieve optimal fat structuring and ice cream meltdown. The net effects of homogenization are in the production of a smoother, more uniform product with a greater apparent richness and palatability, and better whipping ability (1). Homogenization also decreases the danger of churning the fat in the freezer and makes it possible to use products that could not otherwise be used, such as butter and frozen cream.

# 5. Aging

An aging time of 4 hours or greater is recommended following mix processing prior to freezing. This allows for hydration of milk proteins and stabilizers (some viscosity increase occurs during the aging period), crystallization of the fat globules, and a membrane rearrangement, to produce a smoother texture and better quality product (6, 11). Nonaged mix is very wet at extrusion and exhibits variable whipping abilities and faster meltdown (1, 6). The appropriate ratio of solid: liquid fat must be attained at this stage, which is a function of temperature and the triglyceride composition of the fat used, as a partially crystalline emulsion is needed for partial coalescence in the whipping and freezing step, as discussed in Sec. II.B.4. Emulsifiers generally displace milk proteins from the fat surface during the aging period (12, 36, 95), and this is also discussed in detail in Sec. II.B.4. The whipping qualities of the mix are usually improved with aging. Aging is performed in insulated or refrigerated storage tanks, silos, etc. Mix temperature should be maintained as low as possible (at or below 4°C) without freezing.

# B. Dynamic Freezing

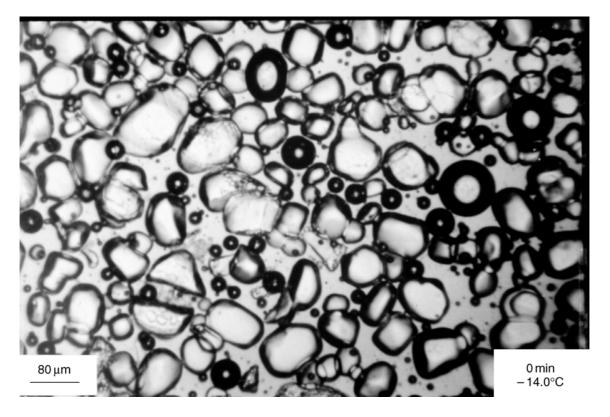
In a continuous scraped surface freezer, numerous processes take place that ultimately influence the overall quality of the ice cream. One of the most important steps, of course, is freezing water into ice. At the same time as ice is being formed, there is also air incorporation, leading to development of air cells and the desired overrun. In addition, destabilization of the fat emulsion (partial coalescence, see Sec. II.B.4) takes place during freezing, which promotes incorporation and stabilization of the air cells. All of these processes take place simultaneously in the minute or less that ice cream spends in the dynamic freezing step. Following this initial phase of ice formation in a dynamic freezer,

where about half of the water is turned into ice, there is a static freezing step, often called hardening (see Sec. II.D).

The mechanisms that lead to ice formation in an ice cream freezer are quite complex. Ultimately, the product exiting the freezer contains numerous small ice crystals. As seen in Fig. 5 (96), the ice crystals in ice cream at the exit of the freezer are somewhat blockshaped and vary in size from a few microns to over  $100\,\mu\text{m}$ . A typical size distribution for hardened ice cream is shown in Fig. 6 (6). The large number of very small ice crystals, estimated to be  $4\times10^9$  crystals per liter (97), gives ice cream its smooth, cool character. The ice crystals must remain below some threshold detection size, often given as about  $50\,\mu\text{m}$  mean size (1), for the ice cream to remain smooth. When crystals become larger than this, the ice cream may be considered coarse. Control of ice crystallization to produce the desired number and size of crystals is critical to producing high-quality ice cream.

# 1. Principles of Ice Crystallization

When ice freezes or crystallizes from any solution, several steps must take place. First, the solution must be cooled below the freezing (melting) point of the solution. The temperature difference between the actual temperature and the freezing point temperature of the mix is the driving force for freezing. Once an appropriate driving force has been attained, formation of the solid ice phase from the liquid molecules must occur. This step is called nucleation, where tiny bits of crystalline ice have just started to form. Once these nuclei begin to form, they continue to grow until some phase equilibrium has been obtained. In freezing, ice continues to form until a thermal equilibrium between the freezing product and its environment has been reached. The total amount of ice that forms



**Figure 5** Ice crystals in ice cream, as observed using light microscopy. (From Ref. 9.)

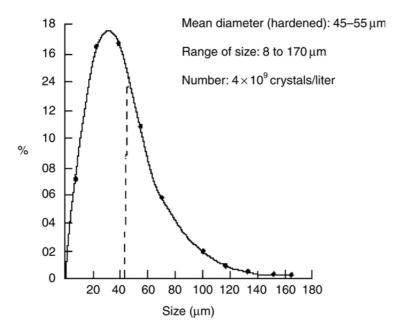
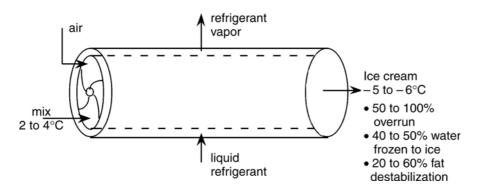


Figure 6 Typical ice crystal size distribution for hardened ice cream. (From Ref. 6.)

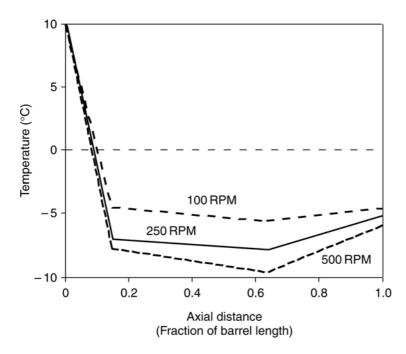
(at any storage temperature) depends on the system. For pure water, all of the water is converted to ice as long as the temperature is below 0°C. In ice cream, however, the other ingredients influence the freezing process and determine how much water turns to ice (the ice phase volume) at any temperature. Both the total amount of ice as well as the nature of the ice dispersion (size, shape, etc.) influence the physical properties of the final ice cream product.

After the product is frozen, the ice phase continues to undergo recrystallization. Recrystallization is a term used for a combination of several events, including melting, growth, and ripening, that occur after the initial ice crystal phase has been developed. Recrystallization leads to changes in the distribution of ice crystals within the system based on the thermodynamic difference in melting point between large crystals and small ones. Typically, recrystallization occurs with no change in ice phase volume.

In continuous ice cream manufacture, mix is pumped into the freezer and flows along the length of the barrel. As the ice cream moves from the inlet to the outlet, ice is frozen, fat is destabilized, and air is injected, as shown in Fig. 7. The mix enters the freezer barrel at a temperature between 0 and 4°C and begins to freeze as it contacts the metal wall



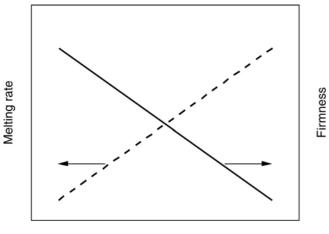
**Figure 7** A schematic diagram to represent inputs and outputs during the continuous freezing of ice cream.



**Figure 8** Axial profile of ice cream temperature as a function of dasher speed within the barrel of a scraped surface ice cream freezer. (From Ref. 98.)

cooled by expanding refrigerant (ammonia or Freon). Ice forms at the barrel wall, since this is where the driving force for freezing is the highest. However, the ice layer that forms is rapidly scraped off of the wall and dispersed into the center of the freezer barrel, where the ice changes form depending on temperature conditions and mixing parameters. As the mix moves axially along the freezer barrel, the amount of ice formed increases as the bulk average temperature of the slurry decreases. At the draw (exit) of the freezer, approximately half the initial water in the mix is frozen into ice, and the product is a pumpable slurry of partially frozen ice cream. The change in temperature along the length of the freezer for a typical ice cream operation is shown in Fig. 8 (98). The final temperature and the amount of ice formed depends on the rate of freezing within the barrel of the freezer. This is controlled by the flow of refrigerant (ammonia or Freon) on the outside of the barrel, the throughput rate of ice cream through the freezer and the type of mixing device used within the barrel of the freezer. In general, conditions in a scraped surface freezer are controlled to give a compromise between the draw temperature (amount of ice frozen) and the stiffness of the ice cream exiting the freezer. The ice cream should be as frozen as possible (since here is where control of ice formation occurs) yet be sufficiently fluid to incorporate inclusions and/or fill the containers without leaving air gaps. This compromise depends to some extent on the type of product being produced and its final form.

a. Phase/State Behavior. Freezing Point Depression. In order for ice to freeze, the temperature of the solution has to be lowered below its freezing point. The temperature at which a solution freezes, or the freezing point, is determined by the concentration and type of solutes present in the mix. The presence of dissolved salts and sugars causes the freezing point of water to be lowered. This freezing point depression occurs because the solute molecules interact with water and inhibit the ability of the water molecules to come together and form an ice crystal lattice (or freeze). The extent of



Freezing point temperature

**Figure 9** Effects of freezing point of ice cream mix on melting rate and firmness of final product. (Based on data from Ref. 99.)

freezing point depression is based on the number of solute molecules and their size. Small molecules have the greatest effect; the higher the concentration of these small molecules, the lower the freezing point. Thus ice cream mixes made with high concentrations of milk salts and lactose, with high sugar content or with high content of low-molecular-weight sweeteners, have low freezing points. For example, use of high-fructose corn syrup as a sweetener gives a lower freezing point (compared to the use of sucrose) owing to the addition of lower molecular weight sugars. Mixes made with high levels of msnf have a low freezing point owing to the addition of milk salts and lactose.

The freezing point of the ice cream mix is an important quality control parameter, since it governs the amount of ice that can form at a given temperature, which affects the quality and textural attributes of the ice cream. As seen in Fig. 9 (99), the melting rate increases and firmness decreases with increasing freezing point (as indicated by osmolality) (99, 100). As the freezing point of the mix goes down (osmolality increases), the ice cream contains less ice and more unfrozen water at any given temperature, which leads to ice cream that is less firm and melts at a faster rate.

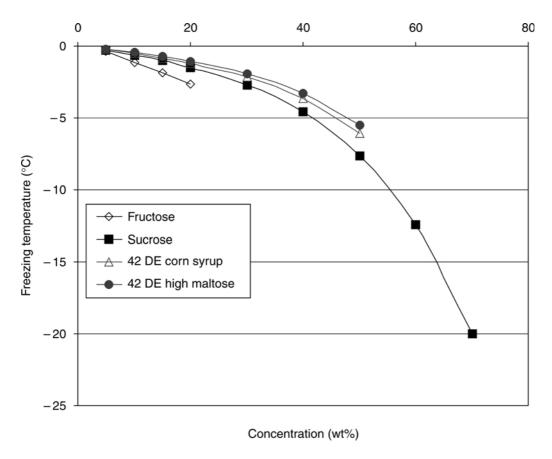
Freezing point depression also can be calculated based on principles of thermodynamics (96), assuming ideal solutions and dilute concentrations. At the point where the two phases (solid ice and liquid water) are in equilibrium, the chemical potentials of the two phases are equal and the following equation can be developed:

$$\frac{\Delta H}{R} \left[ \frac{1}{T_0} - \frac{1}{T} \right] = \ln(X_{\mathbf{w}}) \tag{3}$$

Here,  $\Delta H$  is the latent heat of fusion, R is the ideal gas constant,  $T_0$  is the freezing point of pure water, T is freezing point of solution with mole fraction of water of  $X_w$ . For aqueous foods, Eq. (3) may be modified to give

$$(T_{\rm f} - T_0) = K \frac{C}{MW} \tag{4}$$

where,  $T_f$  is the freezing point (°C) of a solution with concentration C (in g/100 g water),

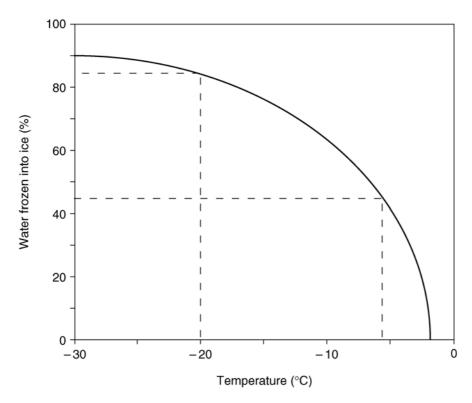


**Figure 10** Freezing point depression curves (freezing temperature as a function of concentration) for several sugars. (From Ref. 96.)

MW is the molecular weight of the dissolved solute, and K is a conversion factor (equal to 1.86 for water).

In simple systems, Eq. (4) gives a good estimate of the freezing point, and it can be used to show the relationship between freezing point and solute content. For example, the freezing point depression curves for several sugars are shown in Fig. 10 (96). Note that fructose has a lower freezing point than sucrose at any equal concentration (wt%) because it has lower molecular weight, and there are more molecules of fructose added (at the equivalent mass of sugar added). Conventional corn syrup solids (42DE), which contains numerous longer chain saccharides, has a higher freezing point than sucrose. In more complex food formulations, the sum of each of the components that impact the freezing point depression is needed. In ice cream mix, it is the combination of sweeteners and milk ingredients used in the formulation that leads to the specific freezing point depression curve for any mix. Sugars (from sweetener and msnf) and salts (from msnf) are the main components that impact freezing point depression of ice cream mix.

Typically, the freezing point depression of an ice cream mix is calculated from Eq. (4) by taking the sucrose equivalents of all the important components that influence the freezing point. Sucrose equivalency values for common sweeteners have been developed (101) for use in ice cream formulations. The contributions of both sweeteners and salts on freezing point are then summed (102) to obtain the initial freezing point of the mix. Equation (4) can also be used to calculate the amount of water frozen into ice for a given ice cream at any temperature by varying the concentration, since freeze concentration of



**Figure 11** Examples of the approximate amount of water frozen into ice for ice cream of standard formulation at given temperatures, based on an equilibrium freezing curve for that formulation. (Based on Ref. 103.)

the unfrozen phase occurs during freezing. Based on the approximate freezing point depression curve and the assumption of slow freezing, the amount of water converted to ice at any temperature can be calculated by a mass balance. For a typical ice cream, a relationship between temperature and the amount of water frozen into ice is obtained, as shown in Fig. 11 (103). Since Eq. (4) technically only works for dilute, ideal solutions, it does not apply very accurately at higher concentrations found in the unfrozen phase of ice cream. Thus correction factors have been developed based on experimental data for frozen sucrose solutions (104).

To calculate the freezing point of a given mix, the effects of sweeteners and salts must be summed. The effects of sweeteners are obtained by summing the contributions of sucrose, lactose (from msnf), and any corn syrups added. For an ice cream mix containing only sucrose, Eq. (5) is used (1).

$$SE_{sw} = \frac{[(msnf \times 0.545) + S]100}{W}$$
 (5)

Here,  $SE_{sw}$  is the sucrose equivalence from sugars; S is sucrose content, W is water content (100 – total solids, %) and 0.545 is the percentage of lactose typically found in msnf. To obtain the freezing point depression associated with this level of sugars,  $FPD_{sw}$ , Table 9 is used (1). The contribution to freezing point depression from salts in msnf is found from Eq. (6).

$$FPD_{sa} = \frac{msnf \times 2.37}{W} \tag{6}$$

**Table 9** Freezing Point Depression in Sucrose Equivalents

	Freezing points	
Sucrose equivalent (%)	(°C)	(°F)
0	0.00	32.00
5	-0.42	31.25
10	-0.83	30.50
15	-1.17	29.90
20	-1.50	29.30
25	-2.08	28.25
30	-2.67	27.20
35	-3.58	25.55
40	-4.39	24.10
45	-5.69	21.75
50	-7.00	19.40

Source: Ref. 1.

Here,  $FPD_{sa}$  is the freezing point depression (°C) for salts contained in msnf, and the constant 2.37 is based on the average molecular weight of the salts present in msnf. To obtain the freezing point depression of the ice cream mix,  $FPD_t$ , the two contributions are summed.

$$FPD_{t} = FPD_{sw} + FPD_{sa} \tag{7}$$

EXAMPLE PROBLEM 3. Calculate the initial freezing point of an ice cream mix containing 16% sucrose, 12% msnf, and 60% water (40% total solids). First calculate the sucrose equivalents from Eq. (5):

$$SE_{sw} = \frac{[12 \times (0.545) + 16]100}{60} = 37.57$$

Now, find the freezing point depression for this level of sucrose equivalent from Table 9. By interpolation,

$$\text{FPD}_{\text{sw}} = 2.31^{\circ}\text{C}$$

For salts, from Eq. (6):

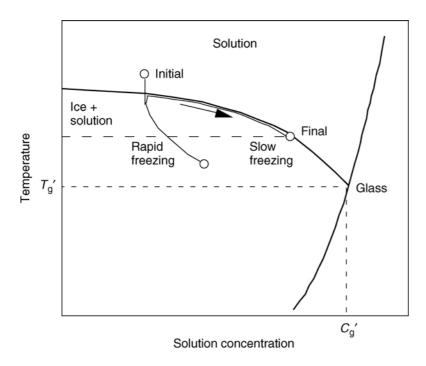
$$\text{FPD}_{\text{sa}} = \frac{12 \times (2.37)}{60} = 0.47 \,^{\circ}\text{C}$$

Find the total freezing point depression for the mix from Eq. (7):

$$FPD_t = FPD_{sw} + FPD_{sa} = 2.31 + 0.47 = 2.78^{\circ}C$$

Thus the initial freezing point temperature for this ice cream mix is  $-2.78^{\circ}$ C.

Freezing Curve. In order for ice to form, the temperature of the system (T) must be below the freezing point  $(T_f)$  of the mix. The extent of subcooling  $(\Delta T = T_f - T)$  determines the rate of freezing, as discussed in the next section. Once freezing occurs, though, several things take place. The change in phase due to the formation of ice causes a release of heat (latent heat of fusion), which increases the temperature in the vicinity of the



**Figure 12** A phase diagram for solutions (e.g., ice cream mix) showing the path of freezing (temperature and solution concentration) for freezing at different rates. Schematic representation of freezing point depression and glass transition curves.  $T'_g$  and  $C'_g$  represent points of maximally freeze concentrated solution. (Adapted from Ref. 96.)

phase change; this heat is removed by the refrigerant. At the same time, removal of water from the mix in the form of ice causes an increase in concentration of the remaining unfrozen phase, which has a lower freezing point due to the higher concentration. Thus, in the vicinity of the ice crystals, the temperature increases and the freezing point decreases. This leads to a freezing profile (Fig. 12) dependent on the rate of freezing (96). For slow freezing, once nucleation starts, the temperature increases to approximately the melting point, owing to the fast release of latent heat, and then begins to decrease as further heat is removed and the concentration increases. Slow freezing results in a freezing profile that essentially follows the freezing point depression curve.

As freezing continues, the unfrozen phase becomes more and more concentrated and the temperature continues to decrease. This leads to an increase in viscosity of the unfrozen phase, until ultimately the viscosity is sufficiently high that the freeze-concentrated unfrozen phase becomes glassy. That is, at some low temperature (the glass transition temperature,  $T_{\rm g}$ ), the unfrozen phase solidifies into a glassy state. Note that this is not a true solid (in the sense of a crystalline solid); rather it is a high-viscosity fluid that acts like a solid for as long as the temperature remains low. The point where the glassy state is formed during slow freezing is called the maximally freeze-concentrated temperature ( $T_{\rm g}'$ ), as seen in Fig. 12. For various ice cream mixes,  $T_{\rm g}'$  has been found to be around -30 to  $-35^{\circ}$ C (85, 105). For slow freezing, the amount of ice formed at any temperature is obtained as described in the previous section, since the system follows the freezing point depression curve. If freezing is very rapid, the temperature and concentration of the solution falls somewhere below the freezing point depression curve,

as shown in Fig. 12. In this case, Fig. 11 no longer applies, and the amount of ice formed at any temperature is less than that shown in Fig. 11 and is dependent on the rate of freezing.

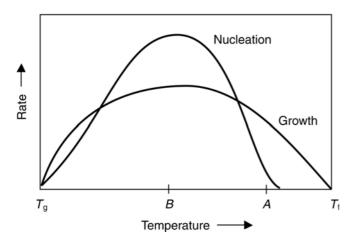
b. Nucleation. The driving force for freezing is the temperature difference between the actual temperature of the system and the freezing (melting) point  $(T-T_f)$ . At higher subcooling, freezing occurs more rapidly; that is, the rate of ice formation is a strong function of the thermal driving force  $(\Delta T)$ . The onset of nuclei formation is the point when the water molecules convert into molecules in an ice crystal lattice. When the temperature driving force is sufficiently high (temperature sufficiently below the freezing point), there is sufficient energy for the water molecules to overcome the energy barrier needed to form an ice crystal surface (the interface between crystal and liquid). Typically, ice formation begins on a surface that catalyzes the formation of ice crystals. This surface may be that of the vessel that contains the solution or particles distributed throughout the solution that provide sufficient energy to order the water molecules in solution and promote nuclei formation. In commercial ice cream manufacture, it is likely that nucleation initially occurs by formation on the metal surface (inner barrel wall) exposed to the refrigerant, since that is where the driving force  $(\Delta T)$  is highest. The rate of nucleation (number of nuclei formed per unit volume per unit time) for melt systems has been described by Eq. (8) (96, 106):

$$J = A \exp\left\{-\frac{BT_{\rm f}^2}{\left(\Delta H_{\rm f}\right)^2 \left(T_{\rm f} - T\right)^2} + \frac{\Delta G_{\rm v}'}{kT}\right\} \tag{8}$$

Here, J is nucleation rate, A is a frequency factor (or preexponential term), B is a constant depending on the solutes present,  $T_{\rm f}$  is freezing (melting) point, k is Boltzmann's constant,  $\Delta H_{\rm f}$  is latent heat of fusion, T is the system temperature, and  $\Delta G_{\rm v}'$  is a diffusion-limited term that describes the mobility of water molecules.

Equation (8) clearly shows the dependence of nucleation rate on operating parameters, particularly the temperature driving force. When the system temperature, T, is close to the freezing point temperature,  $T_f$ , the temperature driving force  $(\Delta T)$  is low and the nucleation rate is low. In fact, at temperatures close to  $T_f$ , nucleation is so slow that the system may effectively remain unfrozen for long times, even though it is below the freezing point of the solution. However, when  $\Delta T$  is sufficiently high, or when the system temperature falls sufficiently below  $T_f$ , Eq. (8) predicts a sudden onset of nuclei formation. As the driving force  $(\Delta T)$  increases, the rate of nuclei formation increases precipitously, giving rise to the spontaneous nature of freezing once it has initiated. When  $\Delta T$  increases to too high a value, the nucleation rate once again decreases owing to the limited mobility of water molecules. As the temperature goes down, the viscosity increases substantially, until eventually the system becomes glasslike. At this point, the  $\Delta G'_v$  term overwhelms the  $\Delta T$  term in Eq. (8), and the nucleation rate again goes to zero. Thus there is a maximum in the nucleation rate curve, as is shown schematically in Fig. 13 (9).

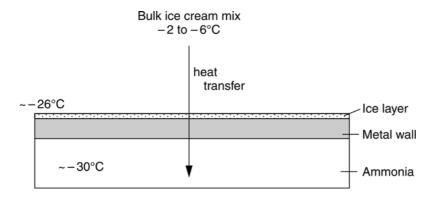
In a commercial scraped-surface freezer, the primary temperature driving force for nucleation occurs at the barrel wall. On the jacket side of this metal wall, liquid refrigerant (either ammonia or Freon) is vaporizing to provide the cooling effect. Vaporizing refrigerant removes heat from the ice cream mix nearest to the barrel wall and creates a high degree of subcooling in the mix at that region (9), as seen in Fig. 14. Ice forms on the metal surface of the barrel wall where the temperature driving force is highest and catalytic nucleation sites exist (microscopic imperfections in the wall itself). Without agitation and scraping, this ice layer would continue to grow and increase in thickness until a thermal equilibrium was attained between unfrozen mix and the coolant.



**Figure 13** Rates of nucleation and growth of ice over the temperature range from the glass transition temperature  $(T_{\rm g})$  to the melting point  $(T_{\rm f})$ . A and B represent temperatures where the nucleation rate is low and high, respectively. (From Ref. 9.)

In commercial freezers, the rotating scraper blades repeatedly clean off the metal surface of the barrel wall. Based on an agitator speed of 200 RPM and a six-bladed agitator, it can be calculated that the metal surface is scraped every 0.05 s. Thus ice has very little chance to build up on the barrel wall. Recent studies (107, 108) using videomicroscopy to observe ice formation on a cooled surface suggest that the scraper blade effectively cleans most of the ice off the metal wall at each scraping. Small pockets or shards of ice left on the wall serve as seeds for subsequent growth of the ice layer between scrapings. These studies suggest that the ice layer initially grows out along the surface to fill an ice layer on the metal wall rather than initially growing out into the solution. Most likely, the scraper blade removes the regrown ice layer before substantial growth into the solution (away from the wall) has occurred. The ice layer that is scraped off the metal wall is dispersed into the bulk mix circulating around the agitators.

The nature of the ice layer scraped off the metal wall in a commercial freezer has been the subject of much discussion in the past decades. Based on work by Schwartzberg (109) and Schwartzberg and Liu (110), it has been suggested that the ice layer in a scraped-



**Figure 14** Approximate driving force  $(\Delta T)$  for freezing of ice cream in a continuous freezer with vaporizing ammonia as refrigerant.

surface freezer forms as dendritic (or needle-shaped) ice crystals extending into the solution (9). The scraper blade then removes these dendrites from the surface and disperses them into the center of the barrel, where subsequent recrystallization and ripening occur. Recent experiments suggest a different form of the ice crystals at the barrel wall. Rather than dendrites extending into the solution, it appears that ice initially grows horizontally along the metal surface, since this is the most favorable direction for heat transfer (107, 108). The ice crystals in this layer are most likely needlelike, although this has not been shown conclusively. Because growth is extremely rapid at the low temperatures of the metal wall, this ice layer is composed of multiple ice crystals surrounded by concentrated mix. Before this layer has a chance to form perfect crystals and exclude solvent molecules from the mix, it is scraped off by the blades and dispersed into the bulk of the freezer. In "slushie" machines that produce iced fruit drinks, the first evidence of ice formation when the refrigeration unit is turned on is thin "flakes" of ice removed from the refrigerated metal surface. Apparently, the scraper blade removes a layer of slush composed of ice and concentrated mix that temporarily maintains its integrity in the bulk, appearing as a thin layer or flake of ice approximately 0.5 to 1 cm in diameter. A submersed microscope in a batch scraped-surface freezer initially catches large (about 250 µm across) sheets of ice that take a hexagonal form (111). Similar forms have been seen growing horizontally along a cooled metal surface (108).

These polycrystalline ice flakes are distributed into the bulk of the freezer by the action of the scraper blades. What happens next depends to some extent on the nature of the bulk phase within the barrel of the freezer. For freezers with open dashers and internal mixers, the ice layer is mixed well with the warmer mix farther away from the refrigerated barrel wall. Here, the blades of the internal dasher can break the ice "flakes" into smaller shreds or pieces. In addition, melting, growth, and ripening take place due to fluctuations in temperature that arise from the heat being removed by the barrel wall and the latent heat associated with melting and growth. A complex heat and mass transfer environment exists in which the ice crystals ultimately grow to product size and shape. Ice crystals exiting the scraped-surface freezer are typically disk-shaped, with sizes ranging from a few microns to over 50 µm. In a closed dasher (one with a high displacement of barrel volume), where the ice cream essentially flows in an annular space between the two cylinders (barrel and dasher), there is much less internal mixing and less opportunity for melting, growth, and ripening. Nevertheless, enough of these processes take place that the ice crystals exit the freezer as disk-shaped crystals (as seen in Fig. 5).

c. Growth, Ripening, and Equilibration. Within the barrel of the scraped-surface freezer, several complex processes related to freezing take place simultaneously. Furthermore, each process affects the nature of the other processes, primarily through influences on heat transfer. The thin layer of polycrystalline ice and slush that is scraped off the barrel wall is colder than the fluid at the center of the barrel. Thus the first thing that happens is that the colder slush flake cools the surrounding environment as it is in turn warmed up. This warming, coupled with mechanical agitation, causes the flake to be broken down into smaller shreds, as has been observed by a submersible microscope in a batch freezing apparatus (111). The polycrystalline ice crystals contained within the slush flakes are dispersed into the bulk solution where they melt, grow, or ripen according to the conditions in their immediate environment. In regions where the temperature is slightly higher than that of the slush from the wall, the ice crystals begin to melt. However, melting takes heat out of the solution as latent heat, which subsequently cools the surrounding environment. The direction of heat transfer determines which regions get the most cooling

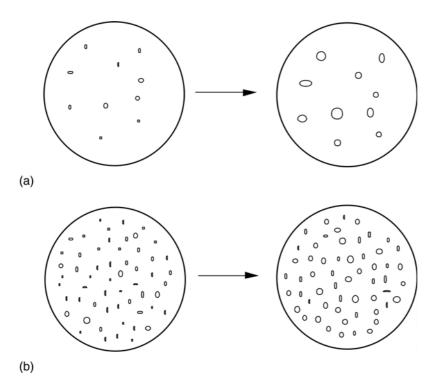
effect. In the regions where the temperature is a little lower than that of the slush from the wall, ice crystals grow owing to the temperature driving force. However, growth causes a release of latent heat, which warms the surrounding environment.

The rate of ice crystal growth is primarily influenced by two mechanisms. Ice crystal growth depends on the rate of counterdiffusion of solute molecules away from the growing interface and on the rate of heat transfer removal from the environment through either the solution or the ice crystal itself (112). The solute molecules present in the ice cream mix (i.e., sugars, salts, proteins, hydrocolloids, etc.) must diffuse away from the growing surface to allow the incorporation of water molecules into the existing crystal lattice structure. The rate of diffusion of these solutes depends on the molecular size (larger molecules diffuse more slowly) and the concentration gradients existing during growth. Once water molecules are incorporated into the crystal lattice, there is a release of the latent heat of fusion, which must be removed by conduction and/or convection mechanisms. In an agitated environment, heat transfer generally occurs most rapidly by convective processes with fluid movement carrying away the heat from the growing crystal surface.

Further complicating these dynamics of melting and growth within the freezer barrel is the thermodynamic mechanism of ripening (112). Ripening is based on the slight difference in equilibrium (e.g., freezing temperature) between crystals of different size. It is well known that very small crystals (less than about 5 µm for ice) have a slightly lower freezing point than large crystals (96). Thus very small crystals may actually melt at the same time (in the same environment) that larger ice crystals continue to grow. In fact, it is this principle of ripening that leads to changes in ice crystals due to recrystallization in storage.

d. Controlling Freezing. The principles of freezing discussed in the previous section are applied in commercial ice cream manufacture to make products with the desired number and size distribution of ice crystals for the highest quality. In the continuous commercial freezer described above, conditions are controlled to maximize the production of numerous small ice crystals. A low-temperature refrigerant (vaporizing ammonia or Freon) is used to lower the temperature of the mix quickly to about  $-25^{\circ}$ C at the surface of the freezer barrel. This low temperature (high-temperature driving force for nucleation) causes nucleation to occur rapidly and results in the formation of many small nuclei. Even though these nuclei ripen and grow as they make their way to the exit of the continuous freezer, they remain quite small (20 to 25  $\mu$ m).

Compare the commercial situation above to that in a small batch home freezer. In both cases, ice forms on a cold metal surface in contact with a refrigerant, with a scraper blade periodically removing the ice layer formed at the wall. In the batch freezer, an ice-brine solution is made to lower the temperature of the ice cream mix. However, this brine reaches temperatures of perhaps only  $-10^{\circ}$  to  $-12^{\circ}$ C. This warmer temperature means that nucleation occurs at a significantly lower driving force than in the commercial freezer (liquid ammonia at about  $-30^{\circ}$ C). According to Fig. 13, the rate of nucleation is significantly lower in the batch freezer, due to the lower  $\Delta T$ , than in the continuous freezer, and thus fewer ice crystals are formed. When the final ice cream products are hardened to the same temperature, the product from the batch freezer, which contained fewer crystals, ends up with significantly larger ice crystals (and potentially coarser ice cream) than the product from the continuous freezer, which had many more smaller crystals. This principle is described schematically in Fig. 15 (113).

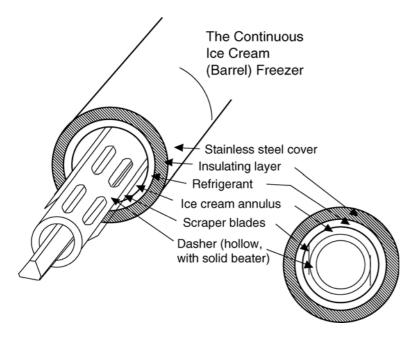


**Figure 15** Schematic depiction of ice crystal size distributions obtained from (a) batch (few nuclei formed) and (b) continuous (many nuclei formed) ice cream freezers, based on nucleation rate. (From Ref. 113.)

#### 2. Operation of the Freezer Barrel

In larger ice cream manufacturing plants, ice cream mix is initially frozen into a semifrozen slurry in continuous freezers. These units are scraped-surface freezers designed to control carefully the ice formation, air incorporation, and fat destabilization. Small-scale operations may utilize a batch freezer, where a single batch of ice cream is frozen at a time. In small soft-serve ice cream and custard stands, batch freezers are sometimes used that involve discontinuous freezing, where ice cream is produced on an as-needed basis.

a. Continuous Scraped-Surface Freezer. A schematic of a commercial continuous freezer is shown in Fig. 16. Ice cream mix at a temperature of 0 to  $4^{\circ}$ C is pumped into the main barrel of the scraped-surface freezer under a pressure of 4-5 atmospheres (3), where it is frozen and aerated at the same time. The pressure inside the barrel is applied to reduce the air phase volume and hence increase heat transfer. Refrigerant is introduced to the outside wall of the annular space between the two concentric cylinders, where vaporization of the refrigerant occurs to provide the refrigeration effect. Heat is removed from the ice cream as it freezes inside the barrel through the walls, to be removed by the vaporizing refrigerant. Typically, either ammonia or Freon, kept at high pressure to maintain the liquid state, is pumped into the freezer, where a lower pressure allows it to expand and vaporize to provide the refrigeration effect. Vaporized refrigerant is removed from the freezer and recompressed in a mechanical refrigeration system. Refrigerant pressure is controlled to maintain the desired temperature (about  $-30^{\circ}$ C) and driving force for heat



**Figure 16** Schematic of the main components of the heat exchanger in a typical continuous ice cream freezer.

transfer removal. The rotating dasher, operating at 150 to 300 RPM within the freezer, holds scraper blades that contact the metal wall and scrape away the slush freezing on the inside of the barrel wall.

As the mix enters the freezer barrel, several things take place at the same time: water freezes in the mix, air is incorporated, and the fat emulsion becomes partially coalesced. Control of these multiple factors is necessary to make ice cream with the desired physical and sensory characteristics. As discussed in the previous section, the freezing of water occurs in the barrel, and the control of ice crystal formation is critical to product quality and shelf life. Since the mix enters the freezer slightly above its freezing point, sensible heat must be removed to lower the temperature to the point where nucleation occurs. This occurs first at the barrel wall with vaporizing refrigerant separated from the ice cream mix by only a thin layer of metal. At the wall, the mix is quickly cooled below the freezing point and nucleation occurs at the barrel wall. It has been estimated that the temperature just on the inside of the barrel wall is about  $-26^{\circ}$ C, based on heat transfer resistances of the metal wall and perhaps a thin layer of ice on the inside of the barrel wall (9). Since the initial freezing point of the mix is about  $-2^{\circ}$ C, there is a significant driving force  $[(-2)-(-26)=24^{\circ}C]$  for nucleation at the wall, and freezing occurs rapidly. Since the refrigerant temperature is maintained along the length of the freezer, the temperature at the barrel wall along the length of the freezer does not change significantly. That is, temperature just at the inside of the barrel wall is likely to be close to  $-26^{\circ}$ C along the length of the entire freezer barrel.

In the center of the barrel, however, the mix temperature is quite different from at the wall, and a temperature gradient in the radial direction exists. Temperature in the center of the barrel may remain above the freezing point for some time as the mix works its way from the inlet to the outlet of the freezer. Eventually, as more and more ice scraped from the wall is mixed in with the warmer mix at the center of the barrel, the temperature in the center gradually decreases. It is at the center of the barrel where melting, growth,

and ripening occur, as discussed in the previous section. Thus the temperature at the center is essentially adiabatically controlled, based on the complex interactions (melting, growth, ripening, etc.) that take place.

It is thought that the decrease in temperature along the length of the barrel at the center of the freezer follows approximately the freezing point depression curve as more and more water is removed in the form of ice (9). Russell and coworkers (98) measured the temperature profile along the length of an experimental freezer and found that temperature decreased rapidly initially (near the inlet), decreased more slowly in the middle section and then increased slightly toward the outlet of the freezer, as seen in Fig. 8 (98). At higher (500) dasher RPM, the temperature decreased to a greater extent than at lower (100) dasher RPM, which suggests that convective mixing from the colder environment near the wall is better with a higher agitation rate. However, the mechanical energy input at the wall of the freezer with a higher agitation rate decreases the efficiency of nucleation and leads to ice cream with larger mean ice crystal size (98). There was a slight increase in temperature of the ice cream just prior to the end of the barrel, where the ammonia jacket ended and no longer provided a cooling effect. This indicates that the ice cream within the freezer barrel was slightly subcooled below the freezing point, and the release of latent heat at the end of the freezer caused the temperature to go up slightly. Once the ice cream was removed from the freezer, however, no temperature changes were observed when the ice cream was held adiabatically. This indicates that no additional crystallization took place once the ice cream was removed from the freezer and suggests that ice cream as it exits the freezer is at a point nearly on the freezing point depression curve for that temperature. Thus estimates of the amount of water frozen into ice at any temperature that are based on freezing along the freezing point depression curve are essentially correct.

The importance of surface nucleation of ice at the barrel wall was shown by attempts to promote nucleation of ice through addition of ice-nucleating bacteria in a commercial continuous ice cream freezer (114). Ice-nucleating bacteria (*Pseudomonas syringae*) were added to an ice cream mix, and the mix was frozen under typical operating conditions in a pilot plant freezer. The ice crystal size of the ice cream exiting the freezer was identical for the control mix and the mix containing the ice nucleator. That these nucleators promote nucleation in the bulk solution suggests that the rate of ice nucleation at the wall of the freezer barrel was so high that the presence of ice nucleators had no effect on the total number of crystals formed in the freezer.

At the same time that freezing is taking place within the barrel, changes are also occurring to the lipid phase and air component. In commercial scraped-surface freezers, filtered compressed air is injected under pressure through a diffuser at the end of the barrel where the mix is input (3). The fine air bubbles formed in the diffuser are incorporated within the mix as the dasher rotates within the barrel. The air cells are broken down into smaller and smaller bubbles based on the shear forces within the freezer as the ice cream is formed (115). Dispersion of air into fine bubbles (about 20 µm in size after draw) requires that freezing occur at the same time to increase the shear forces within the freezer. Whipping air into ice cream mix without freezing results in lower amounts of overrun incorporated and larger air bubble sizes (115).

The fat emulsion also undergoes important changes in the barrel of the scraped-surface freezer (see Sec. II.B.4). Emulsifiers are added to the ice cream mix to decrease the stability of the emulsion droplets and allow partial destabilization during freezing. The shear forces within the freezer result in breakdown of the fine ( $< 3 \,\mu m$ ) emulsion droplets and lead to partial coalescence of the fat globules. In this case, partial coalescence of the

emulsion results in clusters of fat globules that are attracted to the air-serum interface. These partially coalesced fat globules provide stabilization to prevent the coalescence of the air cells so that many small air bubbles remain intact within the ice cream. It is this network of clusters of fat globules that provides meltdown resistance to the finished ice cream.

The refrigeration effect needed for ice cream freezing has been estimated by treating the distinct phases of the freezing process (116). The total energy required may be estimated as the sum of the energy required to cool the mix from the initial temperature to the freezing point, the energy associated with the latent heat needed to convert a certain amount of water into ice, and the energy needed to cool the slush to the draw temperature (1). Although this approach gives only an approximation of the true refrigerant requirements for freezing ice cream, based on the simplifying assumptions, the values obtained give a starting point for calculating refrigeration load in an ice cream facility.

b. Batch Freezer. Operation of a batch freezer proceeds in somewhat the same manner as for a continuous freezer, with several notable differences. That is, similar events take place in batch freezing as just described for continuous freezing, with the ice cream remaining in one place rather than moving along the length of the freezer barrel as in a continuous freezer. One notable difference in batch freezing is that there is typically a lower ratio of heat transfer surface to volume of ice cream. Thus the heat transfer is generally not as efficient in batch freezers as in continuous freezers. Another typical difference between continuous and batch freezing is the nature of the refrigerant used. In commercial batch freezers, as found for soft-serve or custard-type freezers, vaporizing Freon may be used to provide the refrigeration effect. In this case, the temperature differential at the wall of the freezing cylinder may be as low as those found in continuous freezers. Hence very small ice crystals are formed at the wall, scraped off by the mixing blades, and then dispersed into the mix at the center of the cylinder. The temperature profiles at the wall and center of the freezing cylinder are very similar to those found in continuous freezers, except that the temperature changes with time during freezing. When the temperature of the bulk of the ice cream reaches the desired draw temperature, or when the consistency of the ice cream within the barrel reaches some preset or desired value, the ice cream is drawn from the freezer. Typically, draw temperatures from batch freezers are similar to those in continuous freezers. However, due to the quantity of mix to freeze, the residence time required to achieve this draw temperature is much longer than in the continuous freezer, typically 15-30 minutes compared to approximately 1 to 2 min, and the resulting slower rates of freezing result in more recrystallization events in the barrel, larger crystal sizes, and slightly coarser texture when first frozen.

Another significant difference between batch and continuous freezing involves the nature of air incorporation. In batch freezers, the mix is allowed to whip at atmospheric pressure. Hence whipping properties of the mix are very important, and overrun is more variable, being controlled simply by the headspace remaining after the mix charge is put into the barrel. In the continuous freezer, air is injected through controlled valves, so whipping properties of the mix are perhaps less important, and overrun control is exact. Air distribution occurs under pressure in the continuous freezer, and it is the rapid expansion of the air bubbles at draw that establishes the air bubble interface.

Soft-serve ice cream freezers contain a swept-surface barrel freezer similar to the batch freezer, but they also contain a mix hopper that permits the entry of a charge of mix each time a portion of the semifrozen ice cream is removed. Thus the complete barrel is only emptied on shutdown. The air handling systems of some large installation soft-serve

ice cream freezers are a hybrid between batch and continuous freezers, in that the air inlet and barrel itself are pressurized to allow more exact control of overrun.

#### 3. Overrun Calculations

Overrun is the industrial calculation of the air added to frozen dessert products, and it is calculated as the percentage increase in volume that occurred as a result of the air addition. The following examples will show calculations of overrun by volume and by weight, without and with the addition of particulates, and they will also show calculations of target package weights. When packages are being filled on a processing line, package weights should be closely monitored. Deviations can be attributed to variations in the fill level of the package (packaging machine adjustment), variations in the ratio of ice cream to particulate addition (ingredient feeder or ripple pump adjustment), or variations in the overrun of the ice cream (freezer barrel adjustment).

To determine the manufacturing overrun by volume, no particulates, use the equation for overrun determination of a production run, based on the definition of overrun as above, as follows:

% Overrun = 
$$\frac{\text{Vol. of ice cream produced} - \text{Vol. of mix used}}{\text{Vol. of mix used}} \times 100\%$$
 (9)

Example: 500 L mix gives 980 L ice cream, using Eq. (9):

$$\frac{980 - 500}{500} \times 100\% = 96\% \text{ Overrun}$$

Any flavors added such as chocolate syrup in the next example that become homogeneous with the mix can incorporate air and are thus accounted for in the following way.

Example: 80 L mix plus 10 L chocolate syrup gives 170 L chocolate ice cream, using Eq. (9):

$$\frac{170 - (80 + 10)}{(80 + 10)} \times 100\% = 88.8\% \text{ Overrun}$$

Determining manufacturing overrun by volume, with particulates:

Example: 40 L mix plus 28 L pecans gives 110 L butter pecan ice cream, using Eq. (9):

$$110 - 28 = 82 L$$
 actual ice cream surrounding the nuts

% Overrun = 
$$\frac{\text{Vol. of ice cream - Vol. of mix used}}{\text{Vol. of mix used}}$$
  
=  $\frac{82 - 40}{40} \times 100\% = 105\%$ 

The pecans do not incorporate air. This type of determination might be useful if, for example, defects in a given mix were known to show up at >115% overrun. Otherwise, in a calculation of total output, a calculation similar to the one above, with no particulates, may be more useful.

Determining package overrun by weight, no particulates:

% Overrun = 
$$\frac{\text{wt of mix} - \text{wt of same vol. of ice cream}}{\text{wt of same vol. of ice cream}} \times 100\%$$
 (10)

Must know density of mix (wt of 1 L), usually 1.09 – 1.1 kg/L (see example below).

Example: If 1 L of ice cream weighs 560 g net weight (exclusive of package), assuming a density of 1.09 kg/L, using Eq. (10):

% Overrun = 
$$\frac{1090 - 560}{560} \times 100\% = 94.6\%$$
 Overrun

Determining package overrun by weight if the ice cream has particulates in it gives very little information, because both the ratio of ice cream to particulates and the air content of the ice cream affect the final weight.

Determining mix density: The density of mix can be calculated as follows:

$$\frac{\text{wt per liter of water}}{\frac{\% \text{ fat}}{100} \times 1.07527 + \left(\frac{\% \text{ total solids}}{100} - \frac{\% \text{ Fat}}{100}\right) \times 0.6329 + \frac{\% \text{ Water}}{100}} = \text{wt/L mix}$$
(11)

Example: Calculate the weight per liter of mix containing 12% fat, 11% msnf, 10% sugar, 5% corn syrup solids, 0.30% stabilizer, and 38.3% total solids, using Eq. (11):

$$\frac{1.0\,\text{kg/L}}{0.12\times1.07527+(0.383-0.12)\times0.6329+0.617}=1.0959\,\text{kg/L of mix}$$

To determine target package weights, no particulates use the formula

Weight of given vol. of ice cream = 
$$\frac{\text{wt of same vol. of mix}}{\left(\frac{\text{Desired overrun}}{100} + 1\right)}$$
 (12)

Example: Desired 90% overrun, mix density 1.09 kg/L, using Eq. (12):

Net wt of 1 L = 
$$\frac{1.09 \text{ kg}}{\left(\frac{90}{100} + 1\right)}$$
 = 573.7 g

Also, the density of ice cream can be calculated in a similar manner from Eq. (12):

Density of ice cream = 
$$\frac{\text{density of mix}}{\left(\frac{\text{Overrun}}{100} + 1\right)}$$

Example: Density of mix 1100 g/L,

@ 100% Overrun, density of ice cream = 
$$\frac{1100 \text{ g/L}}{\left(\frac{100}{100} + 1\right)} = 550 \text{ g/L}$$

Figuring target package weights, with particulates:

Example: Ice cream with candy inclusion; density of mix 1.1 kg/L; overrun in ice cream 100%; density of candy 0.748 kg/L\*; candy added at 9% by weight (i.e., 9 kg to

<sup>\*</sup> *Note*: density of particulate pieces containing void spaces must be determined by first crushing the material to eliminate void spaces, given that ice cream will fill in the voids after incorporation.

100 kg final product). In 100 kg final product, we have

9 kg of candy 
$$\left(\text{or} \frac{9 \text{ kg}}{0.748 \text{ kg/L}} = 12.0 \text{ L}\right)$$

91 kg of ice cream  $\left(\text{or} \frac{91 \text{ kg}}{\frac{1.1 \text{ kg/L}}{\left(\frac{100}{100} + 1\right)}} = 165.4 \text{ L}\right)$ 

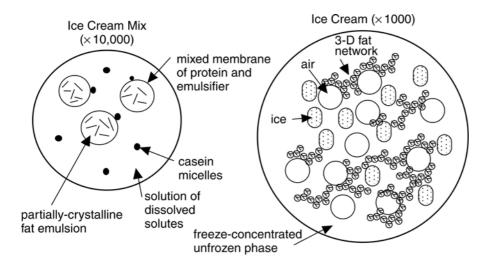
So 100 kg gives a yield of 12 + 165.4 = 177.4 L

1 L weighs 
$$\frac{100 \text{ kg}}{177.4 \text{ L}} = 564 \text{ grams}$$

In many cases, ice creams of different flavors are manufactured to provide the same weight per package for the consumer. As a result, overrun of the actual ice cream in the product varies from flavor to flavor, depending on the density and addition ratio of the particulate ingredients.

#### 4. Fat Destabilization and Foam Stabilization

The texture of ice cream is perhaps one of its most important quality attributes. It is the sensory manifestation of structure; thus establishment of optimal ice cream structure is critical to maximal textural quality. While the dynamic freezing process is generally associated with the formation of the ice phase, aeration and agitation during this process are also responsible for the formation of colloidal aspects of structure, viz., the formation of air bubbles and the partial coalescence of the fat into a major structural element (Fig. 17). The colloidal structure of ice cream begins with the mix as a simple emulsion, with a discrete phase of partially crystalline fat globules surrounded by an interfacial layer composed of proteins and surfactants (Fig. 18). The continuous serum phase consists of the unadsorbed casein micelles in suspension in a solution of sugars, unadsorbed whey



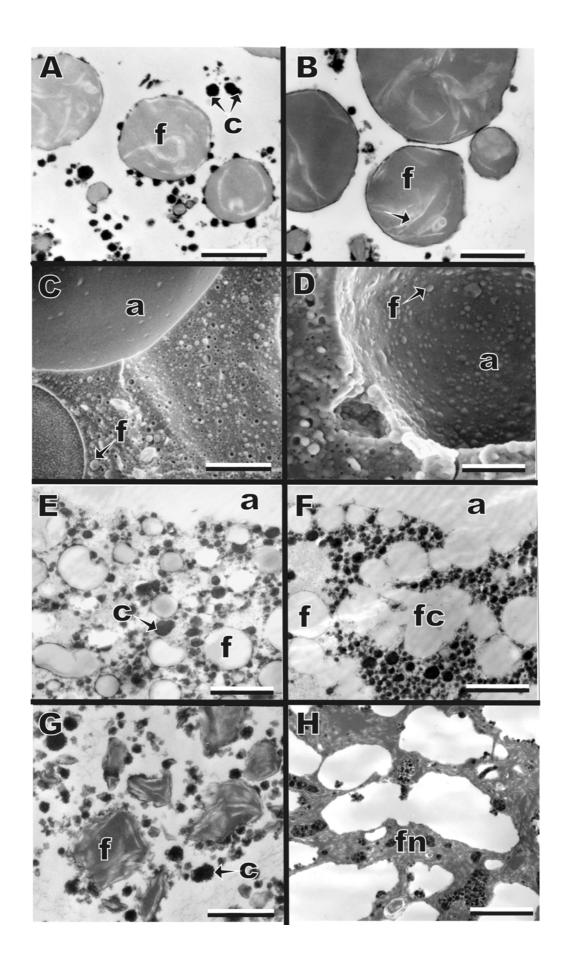
**Figure 17** A schematic representation of the structure of ice cream mix and of ice cream.

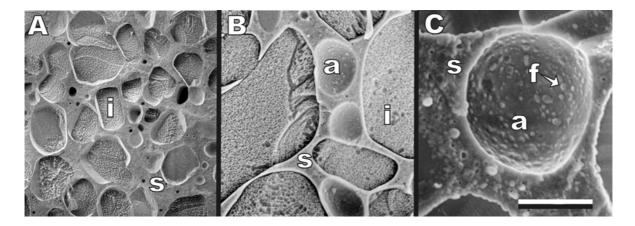
proteins, salts, and high molecular weight polysaccharides. During the "freezing" stage of manufacture, the mix emulsion is foamed, creating a dispersed phase of air bubbles, and is frozen, forming another dispersed phase of ice crystals (Fig. 19). Air bubbles and ice crystals are usually in the range of 20 to 50 µm and are surrounded by a temperature-dependent unfrozen phase (60). In addition, the partially crystalline fat phase in the mix at refrigerated temperatures undergoes partial coalescence during the concomitant whipping and freezing process, resulting in a network of agglomerated fat, which partially surrounds the air bubbles and gives rise to a solidlike structure (Fig. 18) (12, 40, 43, 44, 117).

The development of structure and texture in ice cream is sequential, basically following the manufacturing steps. To describe properly the role of fat in the structure, it is necessary to begin with the formation of the emulsion at the time of homogenization and the role of the ingredients present at the time of homogenization, with particular reference to the fat, proteins, and emulsifiers. After preheating or pasteurization, the mix is at a temperature sufficient to have melted all the fat present, and the fat passes through one or two homogenizing valves. Immediately following homogenization, the newly formed fat globule is practically devoid of any membranous material and readily adsorbs amphiphilic molecules from solution (93). The immediate environment supplies the surfactant molecules, which include caseins, undenatured whey proteins, phospholipids, lipoprotein molecules, components of the original milk fat globule membrane, and any added chemical surfactants (6, 93). These all compete for space at the fat surface. By controlling the adsorbing material present at the time of homogenization, it may be possible to predetermine the adsorbing substances and thus create a membrane with more favorable functional attributes, utilizing natural proteins rather than relying on the chemical surfactants (47). The membrane formed during homogenization continues to develop during the aging step, and rearrangement occurs until the lowest possible energy state is reached (95). The transit time through a homogenization valve is in the order of  $10^{-5}$  to  $10^{-6}$  seconds (91). Protein adsorption or unfolding at the interface may take minutes or even hours to complete (21). It is clear, therefore, that the immediate membrane formed upon homogenization is a function of the microenvironment at the time of its creation, and that the recombined membrane of the fat globule in the aged mix is not fully developed until well into the aging process (12).

Emulsifiers are not needed in an ice cream mix to stabilize the fat emulsion, owing to an excess of protein and other amphiphilic molecules in solution (87,88). If a mix is homogenized without any emulsifier, both the whey proteins and the caseins will form this new fat globule membrane, with the caseins contributing much more to the bulk of the adsorbed protein. However, if added emulsifiers are present, they have the ability to lower the interfacial tension between the fat and the water phases lower than the proteins. Thus they become preferentially adsorbed to the surface of the fat (12, 32, 95). As the interfacial tension is lowered and proteins are eliminated from the surface of the fat, the surface excess (quantity of adsorbed material, mg/m²) is reduced (42), and the actual membrane becomes weaker to subsequent destabilization. This is because the protein molecules, and particularly the caseins, are considerably larger than the emulsifier molecules, so that a membrane made up entirely of emulsifier is very thin (Fig. 18), i.e., there is lower surface excess, although the emulsion stability is thermodynamically favored owing to the lowering of the interfacial tension and net free energy of the system.

Crystallization of fat also occurs during aging, creating a highly intricate structure of needlelike crystals within the globule (Fig. 18). The high melting point triglycerides crystallize first and continue to be surrounded by liquid oil of the lower melting point triglycerides. It has been reported that fat crystallization of emulsified milk fat at





**Figure 19** Low-temperature scanning electron micrographs of the overall structure of ice cream. (A) General overview of spatial distribution of ice crystals (i) within the unfrozen phase (s). Bar (in C) =  $100 \,\mu\text{m}$ . (B) Higher magnification showing air bubbles (a) and ice crystals (i) embedded into the unfrozen serum (s) as discrete phases. Bar (in C) =  $40 \,\mu\text{m}$ . (C) High magnification picture of an air bubble, showing fat globules (f) adsorbed at the air interface and also dispersed in the unfrozen phase (s). Bar =  $20 \,\mu\text{m}$ .

refrigerated temperature reaches equilibrium within 1.5 hours (6). A partially crystalline fat droplet is necessary for clumping to occur. Van Boekel and Walstra (118) found emulsion stability of a paraffin oil-in-water emulsion to be reduced by six orders of magnitude when crystals were present in the dispersed phase. This has been attributed to the protrusion of crystals into the aqueous phase, causing a surface distortion of the globule (118). The crystal protrusions can then pierce the film between two globules upon close approach. As the crystals are preferentially wetted by the lipid phase, clumping is thus inevitable. This phenomenon may account for partial clumping of globules under a shear force. The clusters thus formed actually hold the ice cream serum in their interstices, resulting in the observed dryness. These fat globule chains may also envelop the air cells, thus improving overrun (36), but fat crystals are also known to impair overrun development in whipped cream (21).

The next stage of structure development occurs during the concomitant whipping and freezing step. Air is incorporated either through a lengthy whipping process (batch

**Figure 18** The effect of added emulsifier/adsorbed protein on structure of ice cream mix, ice cream, and melted ice cream. (A, B) Ice cream mix with no emulsifier and with added Polysorbate 80, respectively, as viewed by thin-section transmission electron microscopy. f = fat globule, c = casein micelle, arrow (in B) = crystalline fat, bar = 0.5 μm. (See Ref. 36 for methodology.) (C, D) Ice cream with no emulsifier and with added Polysorbate 80, respectively, as viewed by low-temperature scanning electron microscopy. a = air bubble, f = fat globule,  $bar = 4 \mu m$ . (See Ref. 61 for methodology.) (E, F) Ice cream with no emulsifier and with added Polysorbate 80, respectively, as viewed by thin-section transmission electron microscopy with freeze substitution and low-temperature embedding. a = air bubble, f = fat globule, c = casein micelle, fc = fat cluster, bar = 1 μm. (See Ref. 121 for methodology.) (G, H) Melted ice cream with no emulsifier and with added Polysorbate 80, respectively, as viewed by thin-section transmission electron microscopy. f = fat globule, c = casein micelle, fo = coalesced fat network, fo = coalesced fat network, fo = coalesced for methodology.)

freezers) or drawn into the mix by vacuum (older continuous freezers) or injected under pressure (modern continuous freezers) (1). This process causes the emulsion to undergo partial coalescence or fat destabilization, during which clumps and clusters of the fat globules form and build an internal fat structure or network into the frozen product (1,6), in an analogous manner to the whipping of heavy cream (13). During the initial stages of the whipping of cream, air bubbles have been shown to be stabilized primarily by beta casein and whey proteins, with little involvement of fat (13). Adsorption of fat to air bubbles occurred when the fat globule membrane coalesced with the air-water interface. Only rarely did fat spread at the air-water interface. The final cream is stabilized by a cross-linking of fat globules surrounding each air cell to adjacent air cells, thus building an infrastructure in the foam (119). In skim milk foams, the initial air-water interface is also formed by the serum proteins and soluble  $\beta$ -casein, with little involvement of micellar casein. Micelles become attached as a discontinuous layer but are not deformed or spread (21). It can be postulated that air cell incorporation into ice cream mix follows a similar mechanism. Cross-linking of fat globules from one air cell to the next, thus forming an infrastructure, is less likely due to the reduction in dispersed phase volume from the heavy cream system to the ice cream mix system, but it must also be borne in mind that the air bubbles, fat globules, and aqueous phase are being freeze-concentrated at the same time.

The fat globule clusters formed during the process of partial coalescence are responsible for surrounding and stabilizing the air cells and creating a semicontinuous network or matrix of fat throughout the product, resulting in the beneficial properties of dryness upon extrusion during the manufacturing stages (aids in packaging and novelty molding, for example), a smooth eating texture in the frozen dessert, and resistance to meltdown or good stand-up properties (necessary for soft serve operations) (6, 120). Fat destabilization is enhanced by the emulsifiers in common use (12, 88). When the emulsion is subjected to the tremendous shear forces in the barrel freezer, the thin membrane created by the addition of surfactant is not sufficient to prevent the fat globules from colliding and coalescing, thus setting up the internal fat matrix (36). If an ice cream mix is subjected to excessive shearing action or contains too much emulsifier, the formation of objectionable butter particles can occur as the emulsion is churned beyond the optimum level. Polysorbate 80, having a small molecular weight and producing the lowest interfacial tension compared to mono- and diglycerides, displaces more protein, resulting in a very thin membrane and thus produces the maximum amount of fat destabilization (36).

The extent of fat destabilization can be quantified in several ways. It is sometimes presented as a percentage change in turbidity as measured by a spectrophotometer on diluted samples of mix and ice cream (12). It can also be determined from a solvent extraction technique using a mild solvent, since coalesced fat becomes increasingly susceptible to extraction, whereas emulsified fat does not (95). As well, it can be presented as a change in size distribution of fat globules as measured by laser light scattering techniques (e.g.,  $\% > 3 \,\mu m$ , since 0% of the mix emulsion was greater than  $3 \,\mu m$ ) (42).

Gelin and coworkers (37) demonstrated through light scattering measurements of fat globule size distribution and aggregation that the freezing step is responsible for considerable fat aggregation. This aggregation is initially reversible through dissociation with SDS, but not after fat crystal sintering has occurred. They have also shown the changes occurring to the protein distribution between the aqueous and adsorbed states. It was obvious from their study that the homogenization step accounted for a large amount of adsorbed protein, and that casein was preferentially adsorbed over the whey proteins. The aging and freezing—hardening—thawing steps each accounted for subsequent protein desorption, again mostly of the caseins. The sequential process of partial coalescence

during ice cream freezing has also been examined (12). The incorporation of air alone, or the shearing action alone, independent of freezing, is not sufficient to cause the same degree of fat destabilization as when ice crystallization occurs concomitantly. The freezing process causes an increase in concentration of the mix components, such as proteins and mineral salts, in the unfrozen water phase. It is believed that the ice crystals contribute to the shearing action on the fat globules, owing to their physical shape, and that the concentration of components also leads to enhanced destabilization. However, to create the desired extent of fat destabilization, whipping and freezing must occur simultaneously (87).

Goff and coworkers (121) examined air interfaces in ice cream and fat: air interactions using transmission electron microscopy with freeze-substitution. The structures created by increasing levels of fat destabilization in ice cream (achieved through increased emulsifier concentration in the mix in both batch and continuous freezing) were observed as an increasing concentration of discrete fat globules at the air interface (Fig. 18) and increasing coalescence and clustering of fat globules both at the air interface and within the serum phase (Fig. 18). Air interfaces at the highest levels of fat destabilization were not completely covered by fat globules. It has been suggested that the air interface in ice cream may be covered by a thin layer of nonglobular liquid fat (6). However, there was no evidence of a surface layer of free fat in the work of Goff and coworkers (121). Further, air interfaces in a fat-free ice cream formulation showed a very similar continuous membrane to those from a formulation containing fat, offering further suggestion that the air bubble membrane itself is composed of protein, with discrete and partially coalesced fat globules subsequently adsorbed.

#### C. Flavors and Flavor Addition

Ice cream and frozen dessert manufacturers offer a wide variety of flavors and particulate ingredients to their consumers, which are often the basis upon which consumers make selection choices. Some of the major flavors and flavor categories, based on consumption in North America, are shown in Table 10. Ingredients are added to ice cream in three ways during the manufacturing process: in the mix tank prior to freezing (for liquid flavors, colors, fruit purees, flavored syrup bases, or anything else that will become homogeneous within the ice cream); through a variegating pump (for ribbons, swirls, ripples, revels, etc.); or through an ingredient feeder (for particulates—fruits, nuts, candy pieces, marshmal-

**Table 10** Ice Cream Consumption by Flavor, 2001 Annual, Canada and the US

Flavor	Percentage of production volume		
Vanilla	28.4		
All chocolate	12.6		
Nut flavors	10.4		
Fruit flavors	7.6		
Neapolitan	7.4		
Bakery flavors	5.8		
Candy flavors	3.4		

Source: Data from the International Dairy Foods Association.

lows, cookies and bakery pieces, etc.). In the case of the latter two, this equipment is added in series after the continuous freezer, when the ice cream is already semifrozen. Often, these may be placed in sequence for complex flavors involving multiple components, e.g., a variegating pump and an ingredient feeder or two ingredient feeders. Ingredients added into the semifrozen ice cream should be as cold as possible, either refrigerated or stored at subzero temperatures, so as not to cause any melting and recrystallization of the ice crystals at this point in the process.

Vanilla. Vanilla is the most popular flavor for ice cream in North America. Vanilla ice cream is used to make milkshakes, sundaes, floats, and other types of desserts at the retail level, and it is often an a accompaniment to other desserts, such as cakes or pies. Vanilla is also used in many other flavors where it is a flavor enhancer, e.g., chocolate flavor is improved by the presence of a small amount of vanilla.

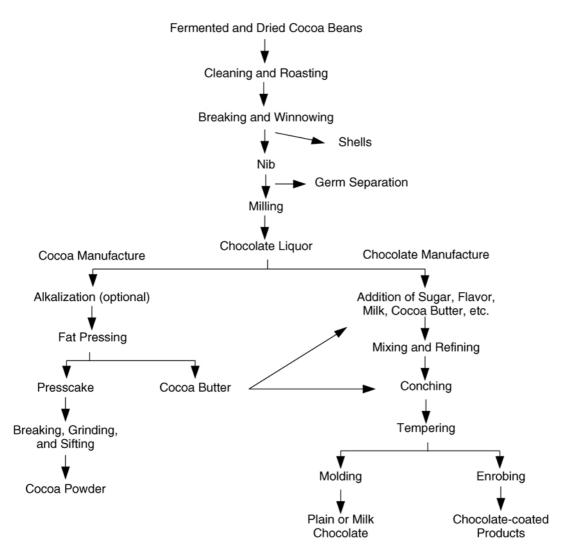
Vanilla comes from a plant belonging to the orchid family called *Vanilla planifolia*, grown typically in Mexico, the islands off the east coast of Africa, particularly Madagascar, Tahiti, South America (Guadeloupe, Dominica, Martinique), and Indonesia (Java). Bourbon beans from Madagascar are often considered the finest and account for over 75% of world production. From each blossom of the vine that is successfully fertilized comes a pod that reaches 15–25 cm in length, picked at 6–9 months. It requires 24–29°C day and night throughout the season and frequent rains with a dry season near the end for the development of flavor. Pods are immersed in hot water to stop biological activity of the seed (which also serves to increase enzyme activity) and then fermented for 3–6 months by repeated wrapping in straw to "sweat" them; then they are uncovered to dry in the sun. Five or 6 kg of green pods produce 1 kg of cured pods. The beans are then aged 1–2 years. Enzymatic reactions during aging produce many compounds, of which vanillin is the principal flavor compound. However, there is no free vanillin in the beans when they are harvested. It develops gradually during the curing period from glucosides, which break down during the fermentation and sweating of the beans. Extraction takes place as the beans are chopped (not ground) and placed in stainless steel percolators. Cold alcohol (no heat involved) and water are pumped over and through the beans until all flavoring matter is extracted. Vacuum distillation takes place for a large part of the solvent. The desired concentration is specified as twofold, fourfold, etc. Each multiple must be derived from an original 10 g beans/100 mL of alcoholic extract.

Vanillin can be and is produced synthetically to a large extent. Vanillin is contained in many types of woods and thus is a by-product of the pulp industry. Compound flavors are produced from combinations of vanilla extract and vanillin. Vanillin may be added at one ounce to the fold for compound flavors. The number of folds plus the number of oz. of vanillin equal total strength, e.g., 2-fold + 2 oz. = 4-fold vanilla-vanillin. However, more than 1 oz. to the fold is deemed imitation. Usage level in the mix is a function of purity and concentration. Typically a single-fold natural vanilla is recommended at 3–6 mL/L mix, a twofold vanilla-vanillin at 2–3 mL/L mix. Some vanillin may improve flavor over pure vanilla extract, so often natural and artificial compound flavors are more desirable than pure natural flavors. Too much vanillin results in harsh flavors.

Chocolate and Cocoa. The cacao bean is the fruit of the tree Theobroma cacao ("Cacao, food of the gods"), which grows in tropical regions such as Mexico, Central America, South America, the West Indies, and the African West Coast. The beans are embedded in pods on the tree, 20–30 beans per pod. When ripe, the pods are cut from the trees, and after drying the beans are removed from the pods and allowed to ferment for 10 days (microbiological and enzymatic fermentation). The beans then are washed, dried, sorted, graded, and shipped for processing. Figure 20 shows a flow diagram for the

processing of chocolate and the manufacture of cocoa. At the processing plant, beans are roasted, the seed coat is removed, and the interior of the bean, called the nib, is ground. Friction melts the fat, and the nibs flow from the grinding as a liquid, known as chocolate liquor. The composition of chocolate liquor is about 55% fat, 17% carbohydrate, 11% protein, 6% tannins, and many other compounds. After the cocoa butter is pressed from the chocolate liquor, the remaining press cake is now the material for cocoa manufacture. The amount of fat remaining determines the cocoa grade: medium fat cocoa, 20–24% fat; low fat, 10–12% fat. There are many types of chocolate that differ in the amounts of chocolate liquor, cocoa butter, sugar, milk, other ingredients, and vanilla. Imitation chocolate is made by replacing some or all of the cocoa fat with other vegetable fats. For ice cream, this provides improved coating properties and enhanced resistance to melting. White chocolate is made with cocoa butter, milk msnf, sugar, but no cocoa or chocolate liquor.

There are two types of cocoa available, American (domestic) and Dutch (alkalized). The latter is treated with an alkali (sodium hydroxide, etc.) to increase the solubility, darken the color, and modify the flavor. The Dutch type is usually preferred in ice cream



**Figure 20** The processing of cocoa into ingredients typically used in chocolate ice cream.

because it gives a darker, less red color, but the choice depends upon consumer preference, desired color (Blackshire cocoa may also be used to darken color), strength of flavor, and fat content of the ice cream (19). For chocolate ice cream manufacture, cocoa is more concentrated for flavoring than chocolate liquor (55% fat) because cocoa butter has relatively low flavor. Hence low-fat cocoa powders are usually utilized at 2–3% (w/w) in the mix. Cocoa is usually added with other dry ingredients at the blending stage, and pasteurized and homogenized with the rest of the mix. Blends of cocoa (2–3%) and chocolate liquor (2%) or chocolate liquor alone (5%) can also be used to produce a chocolate ice cream with enhanced smoothness and with the typical full-fat flavor of chocolate products. Chocolate mixes have a tendency to become excessively viscous, so stabilizer and corn syrup solids content and homogenizing pressure need to be slightly lowered to account for the enhanced viscosity. Sucrose content is generally increased by 2–4% (w/w) in the mix, to offset the slight bitterness from the cocoa.

One frequent defect with chocolate ice cream, particularly soft-serve, is chocolate specking. Cocoa becomes entrapped in partially coalesced fat, which then darkens. Lowering excessive fat destabilization usually alleviates this problem.

Fruit Ice Cream. Fruit flavors are quite popular in ice cream. Fruit for ice cream can be utilized as fresh fruit, raw frozen fruit, "open kettle" processed fruit, or aseptically processed fruit cooked in swept-surface heat exchangers. Fruit additions should use sufficient fruit (15–25% w/w) of choice quality for best fruit ice cream. The more highly flavored the fruit, the less required in ice cream. Fruit should be kept in large pieces in the ice cream where possible, and that is usually a function of the incorporation method. Ingredient feeders are used with continuous freezers to add the fruit pieces or sugared fruit preparations, while a portion or all of the fruit juice, as appropriate when straining of fruit is employed, is added directly to the mix. In the batch freezer, fruit juice is added with the mix at the start of the batch and the fruit pieces are added when the mix has been partially frozen or at draw.

Some small-scale ice cream processors may find it desirable, for a variety of reasons, to use fresh fruit. Such use involves all of the preparation steps of washing, sorting, peeling, destoning, etc. If fresh fruit is being added to ice cream, it should be prepared with sugar in such a way as to allow the sugar to penetrate the fruit. Otherwise, it will freeze to form solid lumps in the ice cream. Sugar draws out juice by osmotic dehydration. If fruits are to be pureed this will not be necessary, although sugar does help to bring out flavor. With strawberries it is advisable to slice in half and treat with sugar at the rate of at least 20–30% sugar, allowing the berries to stand in a cool temperature until sufficient sugar has been absorbed. Sugared fruit can be either strained to separate juice from pulp or cold-stabilized prior to adding to the ice cream, with the use of pectin or starch. In this way, the juice and pulp can be added at the same time through the ingredient feeder.

Fruit for ice cream is usually frozen with the addition of a suitable content of sugar, usually 25–30%. Frozen packs must be thawed before use. Forced thawing with heat will cause rupture of the fruit with resulting poor appearance. Where discrete fruit pieces are not desired in the ice cream, forced thawing may be used. Thawing usually results in juice separation, unless the product has been cold-stabilized with starch or pectin, and if so, this juice should be strained and added to the mix before freezing. Polysorbate 80 (see Sec. I.B.5) is sometimes added to the mix prior to the freezing of fruit ice cream, particularly if the fruit is "wet." This aids in producing a dry ice cream to help incorporate the fruit addition. Depending on the strength of flavor of the fruit preparation and the concentration utilized, it may be necessary to augment fruit flavors with the addition of natural or artificial flavors. Also, sometimes citric acid addition to mix is also desirable.

Fruit can be processed by cooking in a syrup with added sugar to a total sugar content (°Brix) of 50–60% and often stabilized with pectin or starch. This processed fruit moves the problems of procurement, variability, and quality from the ice cream manufacturer to the fruit manufacturer/supplier. The fruit manufacturer can source fruit from around the world and blend it from a variety of sources to achieve year-round supply and consistency. Fruit preparation ensures removal of debris, stones, pits, skins, etc., and cooking ensures microbial safety. By cooking in sugar, the fruit will not freeze as solid in the ice cream and provides a more pleasant texture. For the ice cream manufacturer, this product is available in a ready-to-use form, with no need for thawing, straining, etc., so it involves no product loss. Fruit processed by open kettle methods, however, often provides a cooked flavor that detracts from the natural fruit flavor desired by the ice cream manufacturer and consumer. The processing of such fruit aseptically in scraped surface heat exchangers provides the opportunity to offer an improved flavor and color, a more consistent product, no preservatives, and a longer shelf life.

Variagates. Variagates are injected through a positive pump connected to a small-diameter nozzle or nozzles within the stream of ice cream from the continuous freezer. They are available as a prepared base, e.g., chocolate, butterscotch, marshmallow, strawberry, cheese cake concentrate, etc. and are usually incorporated at 10% (w/w) of ice cream. Almost any flavor can be variegated into ice cream in a variety of contrasting ice cream flavors and colors. A good variegating syrup should not settle out or run into pools in the ice cream. It must not become icy during storage.

Nuts in Ice Cream. Nut-flavored ice creams are also very popular, although concern for consumers with nut allergies has meant strict segregation of nuts from nonnut products and a declaration of possible cross-contamination with nuts, and thus has limited the use of nut flavors in recent years. Nuts should be used in generous amounts, usually around 10% (w/w), and kept in large pieces. Commonly used are walnuts, pecans, filberts, almonds, and pistachios. Brazil nuts and cashews have been tried without much success. Pecans are usually roasted with butter and incorporated into a butter pecan ice cream. Pistachios may be treated in somewhat the same manner as pecans or may be used in the characteristic pistachio ice cream, which is usually colored green and is flavored with bitter almond.

Raw walnuts may be preferred to roasted for flavor, but some form of heat (oven) treatment should be given to walnuts to eliminate surface microbial contamination. Walnuts are often used with a maple flavoring. Almonds are commonly dry roasted to a point just before burning and are added to the mix flavored with vanilla or almond flavoring. Filberts are roasted dry to a light brown color. The skins are removed (blanched) and the nuts reduced in size by chopping. They are added to a mix mildly flavored with vanilla.

Due to potential contamination with extraneous (e.g., shells) and foreign matter, nuts require extensive cleaning and screening. Nuts must be processed in a clean sanitary premise following good manufacturing practices. Nuts should be either oil roasted or heat treated to reduce any bacteria. Microbiological testing for Standard plate count, coliforms, *E. coli*, yeasts, molds, and *Salmonella* sp. is carried out randomly but routinely, and testing for aflatoxin (mold toxin from *Aspergillus flavus*) is performed on peanuts. Nutmeats should be stored either at subzero temperatures in a freezer or at least at 2–4°C to maintain freshness and reduce problems with lipid oxidation in the nuts.

Color in Ice Cream. Ice cream should have a delicate, attractive color that is closely associated with the type of flavoring material that has been added. In some instances, ice cream mix may be slightly colored to give it the shade of the natural product, e.g., 15%

(w/w) fruit produces only a slight effect on color and may need to be augmented. Some fruit solid packs may already be colored by the fruit manufacturer, for convenience to the ice cream manufacturer. Most colors are of synthetic origin and can be purchased in liquid or dry form. Color solutions can easily become contaminated and therefore must be fresh.

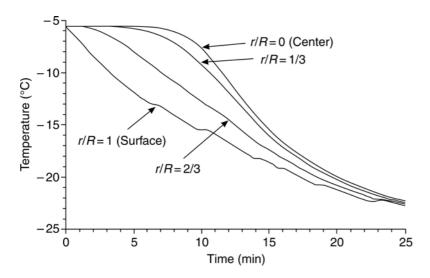
#### D. Packaging and Static Freezing

Once the ice cream exits the freezer as a partially frozen slush, particulate flavors can be added, and then it is pumped into a package, sealed, and hardened. When the semisolid ice cream exits the continuous freezer, it should have the correct stiffness, or ability to flow, for its intended use. For ice cream intended for direct packaging, about half of the water is frozen to ice when the ice cream exits the freezer and it should still be sufficiently fluid to flow and completely fill a package without leaving void spaces. If the draw temperature of the freezer is too low, or the mix is otherwise frozen too much, the ice cream exiting the freezer will be too stiff for proper packaging. In some cases, as for frozen novelties, this high degree of stiffness may be desired so that the ice cream maintains its shape prior to hardening.

Packages of ice cream are sent to a hardening room or tunnel for further freezing. The aim of hardening is to remove heat so that the ice cream cools quickly to temperatures below  $-18^{\circ}$ C. The time required for hardening primarily depends on the size of the package entering the hardening facility and the nature of the refrigeration process within the hardening facility. Very small containers, as in 0.5 L or smaller cups, may take as little as 30 minutes to harden properly, whereas larger bulk-sized containers may take 24 hours. If cartons of ice cream are collected on a pallet prior to hardening, the time for the centermost container to reach hardening temperatures may be substantially longer than 24 hours. Most commercial facilities allow between 12 and 24 hours in the hardening facility to ensure proper freezing.

As the ice cream cools, additional ice freezes in accordance with the freezing point depression curve. It is important to note that typically no new ice crystals (nuclei) are formed during hardening, since the thermal driving forces are generally too small to promote nuclei formation. Thus the increase in ice content (ice phase volume) comes about through a general increase in the size of all existing ice crystals. Clearly the number of ice crystals formed in the initial freezing step will have a big impact on the ice crystal size of the final hardened ice cream. Typically ice crystals increase in size about 10 to 15  $\mu$ m during hardening. That is, the mean ice crystal size after drawing from the continuous freezer may be about 25 to 30  $\mu$ m, but the mean size after hardening is more likely to be between 40 and 45  $\mu$ m.

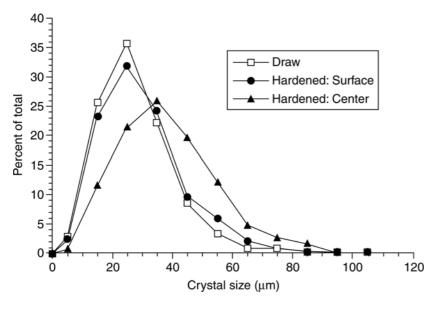
The speed of cooling has a significant impact on the ice crystal size, and this may vary through the container. The ice cream near the outside of the package cools the fastest. The ice cream near the center is insulated by the rest of the ice cream and cools much more slowly. For example, Donhowe (122) followed the temperature decrease at different locations in a half-liter cylindrical container of ice cream during hardening, as shown in Fig. 21 (10). The surface cooled most rapidly, with the center taking nearly 10 minutes even to start cooling. During that 10 minute delay, the ice crystals at the center of the package were undergoing recrystallization at a rapid rate owing to the high temperature. The result is that the ice crystals in the ice cream at the center of the container had substantially larger mean size than the ice crystals in product near the surface, as seen in Fig. 22 (10). This effect becomes even more dramatic when larger containers are hardened. For example, the ice cream at the center of a pallet of containers may remain at elevated



**Figure 21** Temperature profiles as a function of time at different distances from the center (relative radial dimension, r/R) during hardening of a half-liter cylindrical container of ice cream at  $-30^{\circ}$ C (From Ref. 10.)

temperatures for substantially longer than the 10 minutes in this example, and the mean size can get considerably larger. Proper hardening is critical to maintaining the highest quality of the ice cream.

The speed of cooling in the hardening facility also depends on the type of refrigeration system chosen. There are numerous options for hardening ice cream. The choice of hardening facility depends on many factors, including the size of the operation, the types of ice cream products being frozen, and other economic factors. In some cases, as in small operations, the packages of ice cream may simply be transported to an air blast freezer for hardening. In this case, cold air blowing across the packages removes heat from



**Figure 22** Ice crystal size distributions for ice cream at different points within a half-liter container after hardening to -30 °C. Points correspond to container positions in Fig. 21. (From Ref. 10.)

the ice cream as it freezes further. Typically, air at  $-30^{\circ}$ C, cooled by a mechanical refrigeration system, blows past the packages. Good air flow across each individual package is necessary to obtain the fastest rate of cooling.

In larger operations, packages of ice cream are placed on a conveyor (e.g., spiral configuration) and transported through a hardening tunnel to provide rapid convective cooling. The tunnel is maintained at  $-35^{\circ}$  to  $-40^{\circ}$ C and with very high air velocity. The residence time of a package on the conveyor may be between 40 to 160 minutes, which is sufficient to lower the temperature to about  $-18^{\circ}$  to  $-25^{\circ}$ C (1). Again, cold air (-30 to  $-40^{\circ}$ C) blowing across the individual packages provides a rapid rate of cooling in the hardening tunnel. Product exiting the tunnel is then transported to a storage freezer for further distribution.

Another type of hardening system is the plate freezer, which works well for products in containers with flat sides. In the plate freezer, the containers come in contact with a metal surface (the plates) on both sides (top and bottom). The plates are cooled internally with circulating refrigerant so conductive heat transfer is excellent between plates and ice cream. Hardening in a plate freezer can be accomplished in as little as 2 hours (1).

The choice of packaging material is based on many considerations. From a heat transfer standpoint, the package should have sufficiently high heat transfer rate that the ice cream cools rapidly in the hardening facility so that ice crystals are maintained as small as possible. However, during storage and distribution of the ice cream, a good insulating package is desired to minimize thermal fluctuations (and minimize recrystallization during storage). Thus a compromise on type of packaging material used is necessary and often the choice comes down to marketing considerations and the price of the packaging material, with heat transfer and product concerns essentially ignored.

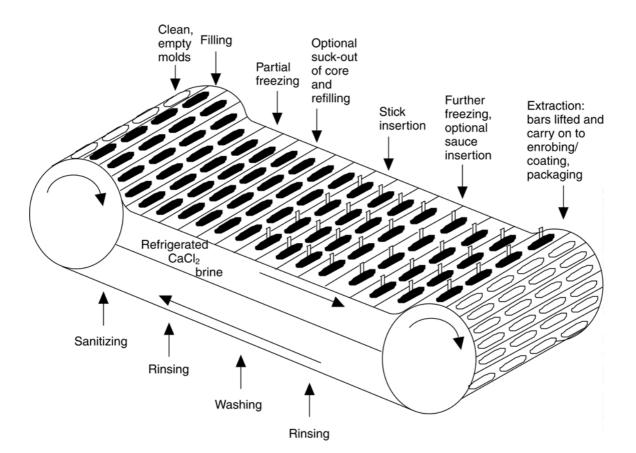
#### E. Novelty/Impulse Product Manufacture

Ice cream products designed for single servings are widely available and are often purchased as handheld items, eaten immediately after purchase. Many of these items are designed specifically for the children's market, so a vast array of shapes exist, and new introductions and variations occur frequently. As a result, this category of products is often referred to either as novelty or as impulse products. They account for a larger share of the ice cream and frozen dessert market in many countries of Europe and Asia than do packaged items designed for home consumption. Examples include stick or stickless bars, cups, and cones. They can be made of many types of frozen desserts, including ice cream with its various fat contents, frozen yogurt, sherbet, puddings, tofu, sorbet, gelatin, and fruit ices. To these are frequently added chocolate baked items such as wafers and cakes, and numerous kinds of fruit. Recent advances in novelty manufacture equipment have greatly increased the number of products available. This equipment is usually high-speed for mass production, but at high capital cost, so production of such items is a specialty market. Strict portion control is a common attribute of modern equipment. Marketing of these items is a large factor in their success.

Novelties can be formed in any of several ways. Most novelty freezing equipment uses ice cream direct from a continuous freezer, at various draw temperatures in order to get the appropriate consistency for the next step. Different configurations of novelty items include direct filling into a preformed single-service cup or edible cone, layering ice cream between biscuits, as in ice cream sandwiches, filling into molds and then quiescently freezing the molds, or extruding ice cream through various shapes or dyes (1).

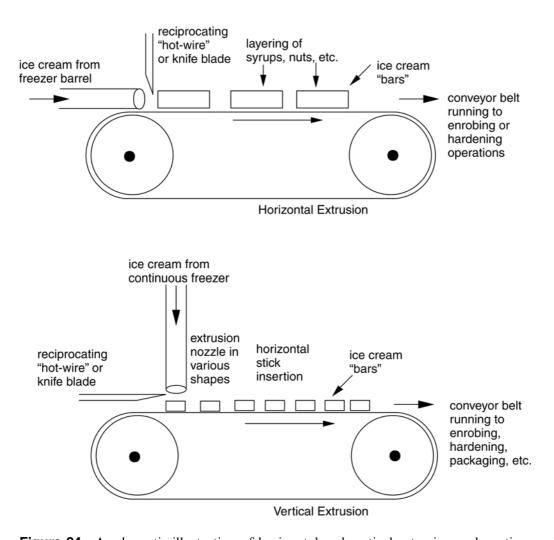
In the molding method (Fig. 23), unfrozen mix, such as juice or fruit ice formulations, or ice cream from the continuous freezer, usually at higher-than-normal draw temperature so it is not too stiff, is transferred to molds that are immersed in or sprayed with chilled brine or glycol. After the product has been partially frozen, sticks are inserted and freezing is completed in the molds. The molds then progress to a section where they are lifted from the secondary refrigerant and briefly exposed to heat (warm brine or water) to loosen the bar, and an extractor picks up the novelty by the stick and passes it to the next station. This station can be an enrober, decorator, or packaging apparatus. Individual packaged items are placed typically in bags or boxes, which may be packed in cartons. Because they typically are very hard when packaged, it is unnecessary to transfer them through a hardening tunnel before sending them to cold storage. Some flexibility with external shapes is possible, but with the use of metal molds, the mold shape must allow for the product to be extracted. Some machines are equipped with flexible molds that peel off the surface of the frozen product during extraction, allowing for more surface features. It is also possible to produce "splits," products with multiple layers from exterior to inner core, on molding machines by filling the mold with the first layer (e.g., fruit ice), allowing for partial freezing of the first layer, then sucking the remainder unfrozen material from the inner core and refilling with another material (e.g., ice cream). In belt-type molding equipment, as in Fig. 23, the molds are then cleaned prior to refilling. Mold freezing equipment is also available in a rotary table-type configuration.

The extrusion method (Fig. 24) involves extraction of ice cream from a continuous freezer at lower-than-average draw temperatures, about  $-6^{\circ}$  to  $-8^{\circ}$ C. The ice cream is



**Figure 23** A schematic illustration of molded novelty freezing equipment.

then pumped through an extruder nozzle and sliced into portions by an electrically heated wire cutter. The extruder may take a horizontal or a vertical form (Fig. 24). The external contour of the slice may be almost any desired shape as is dictated by the shape of the extruder nozzle. By placing different extrusion nozzles inside each other, intricate designs can be formed. Complex extrusions in which multiple flavors or colors are extruded require the use of multiple continuous freezers. Cold-forming or pressing of the extruded item is also possible, allowing complex shapes, designs, patterns, words, etc., to be embossed into the frozen item. If a stick item is desired, the stick is inserted in the extruded ice cream. The pieces are formed on or drop onto carrier plates and pass through a freezing chamber at  $-40^{\circ}$ C with rapid air circulation for fast freezing. Each piece is removed from the carrier plate as it emerges from the freezing chamber. Alternatively, a liquid nitrogen dip can be utilized for rapid setting of surface layers. Portions to be coated with chocolate or other coating are then transferred to an enrober and then through a chill tunnel to set the coating.



**Figure 24** A schematic illustration of horizontal and vertical extrusion and continuous belt-type freezing equipment used in the production of extruded ice cream novelties.

#### F. Storage and Distribution

Once ice cream has been frozen and hardened, it goes through a storage and distribution system designed to get the product to the point of commercial use. This may be a retailer's freezer cabinet and ultimately, in the case of take-home packaging, the consumer's freezer, or it may be another retail outlet like a scooping shop. Whatever the case, the steps and sequence of storage and distribution are critical to maintaining the highest possible quality of the ice cream.

Once the ice cream comes out of the hardening facility, it is typically stored in a low-temperature (-25 to  $-30^{\circ}$ C) freezer within the plant itself until it is shipped to its next destination. It is difficult to generalize the series of distribution points for ice cream, since this depends on many factors, including the size of the ice cream manufacturer, the radius of distribution, and the facilities available. Some companies have their own distribution resources, including refrigerated trucks, whereas other companies must rely on contractors for distribution. In some cases, the ice cream goes first to a central warehouse, whereas in other cases the product may go directly to retail outlets. Everington (123) shows a typical time–temperature history for the distribution of ice cream.

Keeney (124) reported on a survey of ice cream manufacturers and presented some typical time scales for storage at several points in the distribution chain. The time ice cream spent in the factory before shipping varied from 1 to 4+ weeks, with 2 weeks being most common (36%). The next stage in distribution was a warehouse or distribution center, where most companies (64%) reported that the ice cream spent over 4 weeks before being shipped to the point of purchase. The majority of ice cream (68% of respondents) was purchased within 2 weeks at the retail outlet and used within 2 weeks of the consumers' getting the product to their homes. However, in both the retail and consumer stages, some respondents (21%) reported that the ice cream was kept for longer than 4 weeks. Since temperatures are typically more variable in retail outlets and in the consumer's freezer than in the factory or warehouse freezers, ice cream that spends a long time at warmer temperatures is more prone to becoming coarse as the ice crystals continue to get larger by recrystallization.

Ben-Yoseph and Hartel (125) report some typical conditions and storage times at various stages in ice cream distribution, as shown in Table 11. These numbers were obtained from anecdotal reports from various sources and are only meant to indicate the range of conditions that might be found (122). Ben-Yoseph and Hartel (125) used data on

 Table 11
 Approximate Distribution Sequence for Ice Cream

Storage site	Storage time	Mean air temperature (°C)	Fluctuation <sup>a</sup> amplitude (°C)
Manufacturing plant	2 weeks	-22.0	2.0
Distribution vehicle from plant	6 hours	-19.0	2.8
Central warehouse	4 weeks	-24.0	6.0
Distribution vehicle from warehouse	3 hours	-19.0	2.8
Supermarket storage	1 week	-15.6	2.8
Consumer vehicle from supermarket	0.5 hour	21.0	0
Home freezer	1 week	-12.0	2.8

<sup>&</sup>lt;sup>a</sup>Approximate amplitude of temperature fluctuations.

Source: Ref. 125.

the recrystallization of ice cream coupled with rates of heat transfer into a half-gallon container of ice cream to predict the increase in size of ice crystals at various locations within the container (center to surface) as it progressed through the distribution system presented in Table 11. Not surprisingly, the retailer's outlet and the consumer's freezer were two of the most significant sources of quality loss. However, any point of transport from one center to another is cause for concern as temperature spikes (heat shock) due to lack of control can cause significant product damage in a short time.

#### III. PRODUCT QUALITY AND SHELF LIFE

#### A. Flavor Defects

There can be numerous flavor and textural defects associated with ice cream. Excellent reviews on ice cream defects can be found in Refs. (1) and (126). Flavor defects are classified according to origin and include those associated with the flavoring system (lacks fine flavor, lacks flavor, too high flavor, unnatural), the sweetening system (lacks sweetness, too sweet, syrup flavor), the dairy ingredients (acid, salty, lacks freshness, old ingredient, oxidized/metallic, rancid, whey), processing (cooked), and others (absorbed from storage, stabilizer, neutralizer, foreign).

The dairy ingredients give rise to many of the common flavor defects in frozen dairy dessert products. Acid flavors may develop due to microbial growth in the dairy ingredients used in the manufacture of mix or in mix before freezing. However, off-flavor development due to microbial growth is dependent on the type of organisms present. Acidity is developed by lactic-acid organisms, but the organisms that grow at refrigerated temperatures are mostly psychrotrophs, and off flavors associated with their growth are usually fruity and/or bitter in nature, from peptides derived from proteolysis. Salty flavors may arise from formulations that are too high in msnf, especially if whey powder is used. Whey powder tends to be higher in natural milk salts than skim milk powder. However, it should also be recognized that salt is often an ingredient in mix formulations, for flavor enhancement, and too much salt may have been used. Another source of high salt flavor may be salted butter, used in error rather than sweet butter.

Defects in ice cream flavor associated with the fat phase are usually related to either lipolysis of free fatty acids from triglycerides by the action of lipases (known in the dairy industry as rancidity) or autoxidation of the fat resulting in oxidized flavors (oxidative rancidity as distinct from lipolytic rancidity). These defects tend to be present in the raw ingredients used in ice cream manufacture, rather than promoted by the manufacturing process itself. However, similar precautions to the processing of milk must be taken to ensure that these flavor defects are not present.

Oxidation of milk and other fats proceeds by the well known autoxidation reaction in three stages: initiation, propagation, and termination. In milk, the initiation reactions involve phospholipids present in the fat globule membrane. Free radicals formed from phospholipids are then able to initiate oxidation of triglycerides, especially in the presence of copper and proteins (21). During propagation, antioxidant compounds such as tocopherols and ascorbic acid are depleted, while peroxide derivatives of fatty acids accumulate. Peroxides, which have little flavor, undergo further reactions to form a variety of carbonyls, some of which are potent flavor compounds, especially some ketones and aldehydes. Most methods available to monitor lipid oxidation are unsuitable as an early index of oxidized flavor development in milk. Measurement of peroxides is not useful because peroxides are unstable intermediates; tests based on colorimetric reaction of

thiobarbituric acid with malonaldehyde show some correlation to sensory values but are rather insensitive; and direct measurement of oxygen uptake is only suitable for controlled experimental conditions.

Milk may oxidize as a result of factors either extrinsic to the milk or intrinsic to it (21,127). Important extrinsic factors include contamination with metals, temperature of storage, oxygen tension, heat treatment, agitation, light, and acidity. Both copper and iron may catalyze lipid oxidation but probably only copper is significant in milk. Added copper is much more potent than natural copper because a significant portion of added copper goes directly to the fat globule (21). Significant intrinsic factors affecting milk fat oxidation include metalloproteins such as milk peroxidase and xanthine oxidase, endogenous ascorbic acid, which acts as a cocatalyst with copper to promote oxidation, endogenous copper content and endogenous antioxidants, mainly tocopherols. Fresh forage is well known to control spontaneous oxidation, as indicated by obvious seasonal effects on the incidence of oxidized flavor. This effect is probably due to increased levels of endogenous antioxidants.

Hydrolysis of fatty acid esters by the action of lipases results in the common flavor defect known as lipolytic or hydrolytic rancidity and is distinct from oxidative rancidity (127,128). Lipolysis in dairy fats can be extremely detrimental owing to the number of highly volatile short chain fatty acids present, especially butyric acid. Lipases are unique among enzymes in that they are active at the lipid-serum interface. In milk, lipases are ineffective unless the fat globule membrane is damaged or weakened in some way. Lipolysis may be caused by the lipoprotein lipase (LPL) that is endogenous to milk or by bacterial lipases. The properties of the fat globule membrane are most important to lipolysis. Mastitis, which alters milk composition, also increases sensitivity of the fat globule to lipolysis. Other factors that destabilize the fat globule membrane, especially agitation and/or foaming, also promote lipolysis. Lipolysis is accelerated by the replacement of the native membrane with surface active material (mainly casein micelles and whey proteins) from the plasma (128). This effect is at least partly due to redistribution of LPL from the plasma to the fat globule membrane and accounts for greatly increased lipolysis after homogenization. In the milk from some animals, lipolysis may proceed without subsequent thermal or mechanical activation. This effect, frequently referred to as spontaneous lipolysis, is unlikely to occur in herd milks or in pooled milks because it is prevented by mixing affected milk with three to five times its volume of normal milk. The major conditions that influence spontaneous lipolysis are late lactation, insufficient fresh forage, and low-yielding cows.

Cooked flavors in dairy products, including ice cream mix, are caused by using milk products that have been heated to too high a temperature or by using excessively high temperatures in mix pasteurization. The flavor is typified by scalded milk and is caused by sulfhydral groups from denaturation of disulfide bonds in whey proteins. If it is mild, it can dissipate with time as the sulfhydral groups oxidize, so often it is most noticeable directly after heat processing. A mild cooked flavor is not objectionable, but intense heating can cause the defect to linger and become increasingly objectionable.

Ice cream can sometimes absorb off-flavors from its storage environment. Volatile compounds like smoke, ammonia, paint or diesel fumes have been known to be detectable in ice cream after inadvertent exposure to these odors. It is thus important to recognize that storage environments must be kept free of strongly volatile materials.

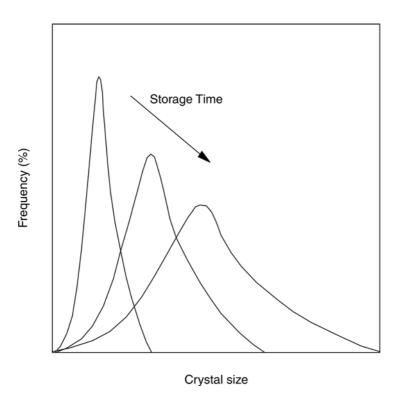
#### **B.** Texture Defects

Considerable effort goes into processing ice cream so that the final product has the desired consumer appeal. From a structural standpoint, this involves controlling ice crystallization, air incorporation, and fat destabilization. During storage, however, significant changes can occur to the structural elements that lead to loss of quality. Textural defects common to ice cream include recrystallization of ice crystals, lactose crystallization (sandiness), and shrinkage.

#### 1. Recrystallization

In ice cream, numerous small crystals are desired for the smooth texture that they impart. Thermodynamically, however, this state is inherently unstable owing to the very high surface area of ice crystals. In principle, this system would be in a lower energy state if the ice phase took the form of a single very large crystal to minimize the surface area (or more correctly, the surface energy). Thus there is a thermodynamic driving force for the small crystals in ice cream to disappear, leaving fewer and larger ice crystals. Recrystallization is seen as an increase in mean size and a widening of the range of sizes (Fig. 25), and it is accompanied by a decrease in the number of crystals (96).

The driving force for this rearrangement is based on the Kelvin equation, which states that the equilibrium temperature of a crystal surface is dependent on its radius of curvature. Thus smaller ice crystals have a slightly lower equilibrium temperature than larger crystals. In a mixture of ice crystals as found in ice cream, the small crystals are less stable than the larger ice crystals. During storage, the smaller ice crystals melt away at the

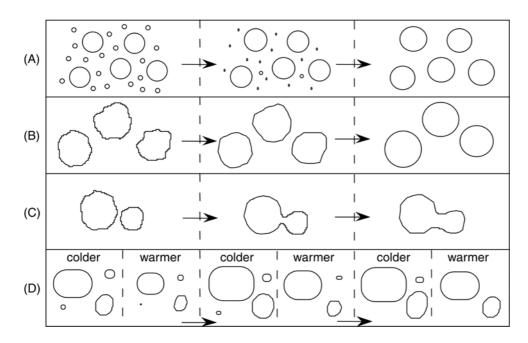


**Figure 25** Typical changes in crystal size distribution during storage. The arrow represents a decrease in the frequency of the crystals found within a size range with increasing mean size. (From Ref. 96.)

same time that the larger ice crystals grow larger, as shown schematically in Fig. 26A. This increase in size of larger ice crystals at the expense of smaller crystals is often called Ostwald ripening, or simply ripening.

However, calculations of the difference in equilibrium temperature between small and large ice crystals in ice cream show that this difference is only significant for very small crystals (10, 122). The difference in driving force, expressed as a difference in equilibrium temperature, between crystals of only 1 µm in radius is less than 0.05°C. For a crystal of 10 µm radius, the temperature difference is less than 0.005°C. Thus the driving force for Ostwald ripening for ice crystals in ice cream is very small. In fact, Donhowe and Hartel (72) did not observe true Ostwald ripening in extensive studies of mechanisms of ice recrystallization during storage of ice cream under accelerated recrystallization conditions on a microscope slide. It was found that other mechanisms were more important in ice cream. Nevertheless, it is this slight difference in equilibrium temperature between large and small crystals that, over long periods of time, can lead to significant changes in the state of ice crystals in ice cream (and other frozen foods).

The main static (constant temperature) mechanisms for the recrystallization of ice crystals during storage include accretion and isomass rounding (10). When the storage temperature is constant, these two mechanisms are responsible for recrystallization of ice crystals in ice cream (72). Isomass rounding is very similar to Ostwald ripening, but it is based on regions of a single crystal with different radii of curvature. A spherical ice crystal would not undergo isomass rounding since the radius of curvature is uniform at all points of the sphere. In other words, a sphere has the minimum surface-area-to-volume ratio. Ice crystals in ice cream are not spherical in nature (see Fig. 5) but have a higher surface-area-to-volume ratio. Ice crystals in ice cream are somewhat irregularly shaped, based on the mechanisms of ice formation in the freezer barrel. Thus there is a driving force for the sharper edges (protruberances) to melt away and for the flatter sides to grow out until the ice crystal approaches a spherical state (Fig. 26B). This process has been observed for ice



**Figure 26** Mechanisms of recrystallization. (A) Ostwald ripening. (B) Isomass rounding. (C) Accretion. (D) Melt–refreeze.

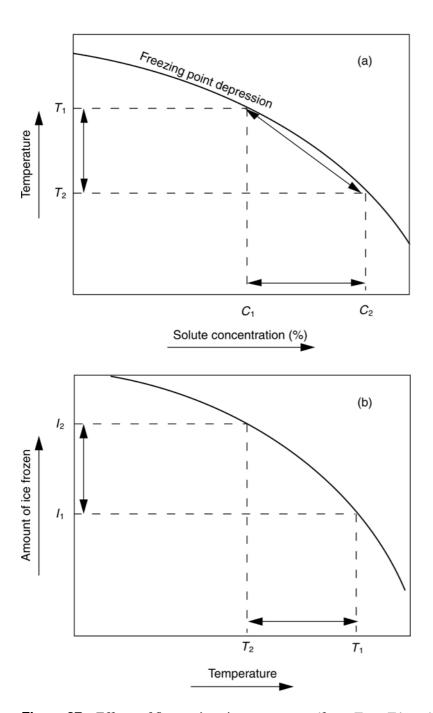
crystals in ice cream held at relatively warm temperatures  $(-5^{\circ}\text{C})$  (72). In this case, the ice crystal dispersion in ice cream progressed from the initial irregular-shaped crystals to essentially spherical crystals over time. Because the driving force for this transition is very small (the differences in size characteristics are very small), the process is slower than other recrystallization mechanisms.

Another important mechanism of recrystallization under constant temperature conditions is accretion. It has been estimated, based on the physical number and sizes of ice crystals and air cells, that ice crystals in freshly hardened ice cream are separated, on average, by a serum film that is less than 10 µm thick (6). This close proximity leads to an instability in the region between the two crystals that leads to bridge formation and eventually to accretion (Fig. 26C). Accretion has been found to be the main mechanism of recrystallization during the initial stages when ice crystals are closely packed together. Once the crystals have become larger and more separated, the importance of accretion diminishes (72, 75).

Although it is informative to understand these static mechanisms for recrystallization, ice cream is rarely (if ever) stored under conditions where temperature is constant. As documented in Sec. II.G, temperatures are continuously changing during storage and distribution of ice cream. Even when stored under "constant" temperatures, most refrigeration systems show some temperature fluctuation as compressors cycle on and off. Thus the process of melting and refreezing is continually occurring, and this process can have a dramatic impact on the ice crystals. In fact, the melt-refreeze mechanism of recrystallization is probably the most important process leading to the change in ice crystals in ice cream during frozen storage (59, 72). As temperature fluctuates in ice cream, the amount of ice (phase volume) changes accordingly. If the temperature fluctuations are relatively slow, the ice phase volume changes according to the equilibrium freezing point depression curve. This can be seen schematically in Fig. 27 (96). When temperature increases, the amount of ice present decreases according to the freezing point depression curve. All ice crystals melt away to some extent, but the smallest crystals melt away a little faster (due to the lower equilibrium temperature) and may eventually disappear (melt away completely). Once a crystal has disappeared, it no longer returns, and no new crystals nucleate (driving force is too low). The mass initially contained in that ice crystal must now be redistributed on the remaining crystals when the temperature is lowered and the ice phase volume increases. This process is seen schematically in Fig. 26D. The meltrefreeze mechanism is the primary mechanism for recrystallization in ice cream under conditions where temperature is changing (59, 72).

The rate of recrystallization in ice cream during storage and distribution is dependent on numerous factors, including the initial state of the ice crystals in the ice cream, storage temperature and fluctuations, and formulation factors (10). Extended shelf life requires that the ice crystals be maintained as small as possible for as long as possible. Of the parameters that influence recrystallization, storage conditions and formulation factors are two of the most important.

The rate of recrystallization is a strong function of temperature, with the rate decreasing significantly as storage temperature decreases (59, 72). Each of the mechanisms of recrystallization described above progresses more slowly as the temperature is decreased. The result is that the rate of recrystallization decreases as storage temperature decreases. In fact, if ice cream is stored below its glass transition temperature, molecular mobility will be sufficiently low that the recrystallization rate will effectively go to zero. The glass transition temperature of ice cream is about  $-32^{\circ}$ C (85, 105). However, the rate of recrystallization typically is quite low if storage temperature is maintained below about



**Figure 27** Effects of fluctuations in temperature (from  $T_1$  to  $T_2$ ) on (a) change in the concentration of the unfrozen phase ( $C_1$  to  $C_2$ ) and (b) change in amount of ice frozen ( $I_1$  to  $I_2$ ). (From Ref. 96.)

 $-20^{\circ}$ C (72). The extent of temperature fluctuations also influences the rate of recrystallization through the effect on the melt-refreeze mechanism. Based on Fig. 26, the effect of temperature fluctuations depends on the storage temperature, since the change in ice phase volume with a given change in temperature decreases as temperature decreases (72). Thus storage at  $-20.0 \pm 2.0^{\circ}$ C has much less effect on recrystallization than storage at  $-8.0 \pm 1.0^{\circ}$ C. A heat shock index can be used to quantify this effect (129).

Since the temperature changes during the various stages of storage and distribution, the rate of recrystallization changes during storage according to the local temperature and fluctuations. Furthermore, different points within a single package experience different thermal conditions and undergo recrystallization at different rates. Donhowe and Hartel (73) showed that ice crystals at the center of a half-gallon container of ice cream remained the smallest, whereas ice crystals near the package surface experienced the greatest rate of recrystallization. The thermal insulating capacity of ice cream, in effect, protects the interior of the ice cream from external temperature fluctuations. Ben-Yoseph and Hartel (125) used typical temperatures and times in different stages of the distribution of ice cream and the rates of heat transfer into a package to predict the ice crystal size at any point in a container of ice cream based on the recrystallization kinetics of Donhowe and Hartel (72). The effects of storage temperatures on ice crystal size at different points in the distribution system were clearly demonstrated.

Of the formulation factors that influence recrystallization, stabilizer and sweetener types are the two most important. In fact, stabilizers are added to ice cream primarily to control recrystallization during storage. However, it is not clear still exactly how stabilizers affect recrystallization (see Sec. I.B.4). Several potential mechanisms have been hypothesized for the effect of stabilizers on recrystallization (10). These include (a) an increase in viscosity of the unfrozen phase, (b) the specific inhibition of ice crystal growth rates, (c) physical obstruction due to the formation of a weak gel structure (58, 71), (d) a change in thermal properties of ice cream due to the addition of stabilizer (82), and (e) a decreased perception of iciness due to the addition of stabilizers (81). It is possible that each of these potential mechanisms plays a role in the effect of stabilizers on recrystallization. However, further work is needed to verify exactly how stabilizers act to inhibit ice recrystallization during the storage of ice cream.

The type of sweetener used in the mix formulation has also been found to influence the rate of recrystallization (74, 84). The effect of sweetener type, however, is primarily related to the amount of water frozen into ice at any temperature. Hagiwara and Hartel (74) correlated recrystallization rates during the storage of ice cream with the calculated amount of water frozen into ice for ice creams made with different sweeteners. Recrystallization rates decreased proportionally as the amount of water frozen into ice increased. Since the amount of water frozen at any temperature is directly related to the freezing point, the recrystallization rate also was seen to decrease as the freezing point temperature increased. Since recrystallization is a diffusion-limited process (based on migration of water molecules), more ice at a given temperature (and less water) leads to slower recrystallization owing to the lower mobility of the water molecules. The lower mobility correlates with an increase in the glass transition temperature of the ice cream (74).

#### 2. Lactose Crystallization

The problem of "sandiness" in some ice creams during storage has been related to the crystallization of lactose from the milk solids in the formulation (1, 130). It is not only that lactose crystals appear in ice cream during storage but that these lactose crystals must grow to sufficient size that they can be detected by the palate and distinguished from ice crystals (131). Based on various sources, it has been estimated that the critical size for lactose crystals in ice cream is about  $15 \,\mu\text{m}$ . Above this size, their presence can be detected as a sandy or grainy characteristic that is different from the coarse texture associated with large ice crystals. When present in ice cream, lactose crystals dissolve at a much slower rate than ice crystals melt. Thus the lactose crystals remain in the mouth even after the ice cream has melted; hence the sandy mouthfeel.

Lactose in ice cream crystallizes when the concentration in the serum phase (unfrozen concentrate) exceeds the solubility concentration of lactose. Since the solubility of lactose is very low (and decreases as temperature goes down), lactose is supersaturated and prone to crystallize at almost any level in ice cream stored at common freezer temperatures. In fact, thermodynamically, lactose should crystallize in just about all ice cream since it is in the supersaturated state at storage temperatures. The fact that lactose does not crystallize in all ice cream during storage may be attributed to the slow kinetics of lactose nuclei formation at these conditions. The viscosity of the unfrozen phase is sufficiently high that lactose nucleation is inhibited for extended periods of time (and may not occur within the shelf life of an ice cream product).

Thus two competitive forces are at work that govern crystallization of lactose in ice cream. The first is the increase in concentration driving force as temperature is decreased, which tends to promote lactose crystallization at lower temperatures. Working against this, however, is the decrease in molecular mobility as the temperature is decreased. Thus there is a storage temperature where lactose crystallization is at a maximum. For a wide range of commercial ice creams, this temperature occurs at about -10 to  $-12^{\circ}$ C (130, 132, 133). Storage in this temperature range leads to the most rapid lactose crystallization in ice cream. Storage at both higher and lower temperatures required longer times for onset of lactose nuclei formation (132).

Of the formulation factors responsible for lactose crystallization, the initial milk solids level in the mix is probably the most important. An upper limit of 15.6 to 18.5% msnf has been suggested to prevent lactose crystallization, with the higher limit for products that move quickly through the distribution chain (1). The presence of sucrose and stabilizers may have an inhibitory effect on lactose crystallization, perhaps through their effect on viscosity of the unfrozen phase during storage. However, addition of powdered or particulate ingredients (e.g., nuts) after initial freezing tends to promote lactose crystallization through two potential mechanisms. Any particulate material added may act as nucleation sites for lactose and promote graining, and it is widely recognized that agitation of a supersaturated sugar solution enhances the likelihood of nucleation (134).

#### 3. Shrinkage

In some situations, ice cream that has been improperly handled exhibits shrinkage: the ice cream pulls away from the walls of the container. Many parameters have been implicated in the mechanism of shrinkage, including formulation factors like improper use of proteins, emulsifiers and stabilizers, and external factors like atmospheric pressure (49). Shrinkage results from a loss of discrete air bubbles as they coalesce and begin to form continuous channels, eventually leading to collapse of the product itself into the channels (48). Shrinkage tends to occur most often after the ice cream experiences a decrease in pressure, as when ice cream is shipped across mountains or transported by plane, which first causes a volume expansion. The extent of air channeling, and hence a measure of ice cream susceptibility to collapse and shrinkage, can be measured by determining the response in volume of the ice cream to pressure changes, given that the volume of discrete bubbles will correlate directly to pressure changes, while the volume of air channels will not (135).

According to the ideal gas law, the size (volume) of an air bubble is related to the external temperature and pressure, assuming that the volume is free to change. As the temperature is decreased, at constant pressure, the volume of an air bubble will decrease.

As pressure is increased, at constant temperature, the air bubble should also contract. For example, when ice cream exits the draw of a continuous freezer, pressure is reduced (pressure within the freezer is higher than atmospheric pressure), and all the air bubbles should expand slightly. At this point, though, the viscosity of the ice cream is sufficiently low that this expansion can easily be accommodated by the surrounding matrix, and the air bubbles approach an equilibrium at atmospheric pressure. Cartons of ice cream are filled to their final weight and volume at this point, and any changes in volume during later storage and distribution may lead to negative changes in the ice cream's appearance. After hardening, when the surrounding matrix has stiffened considerably, subsequent changes in pressure (or temperature) can lead to changes in the forces between the air cells and the surrounding matrix. Expansion or shrinkage, depending on the conditions, may be the result.

Goff et al. (136) reported on the effects of vacuum storage on expansion and shrinkage of ice cream. Containers of ice cream at  $-16^{\circ}$ C were exposed to reduced pressure (8 in. Hg) for 3 hours and then stored for 6 days at  $-16^{\circ}$ C. Volume changes were measured 3 hours after release of vacuum and again at the end of 6 days of storage. Expansion of the ice cream was observed after the vacuum storage, in accordance with the ideal gas law.

However, after 6 days of storage, those same ice creams exhibited shrinkage. In all cases, ice creams made with higher overrun had the greatest expansion and subsequent contraction. At  $-16^{\circ}$ C, the unfrozen matrix must still be sufficiently pliable that a change in atmospheric pressure can cause a change in volume of the ice cream. Interestingly, although the period of vacuum exposure caused expansion, the ultimate result when pressure was brought back to atmospheric was shrinkage of the ice cream volume. This suggests that the unfrozen matrix expanded with the increased air bubble size initially and then relaxed to a smaller volume than originally found. Goff et al. (136) related this to the nature of the interface between the air bubble and the unfrozen serum. They suggested that components like proteins, stabilizers, and emulsifiers play an important role in determining the viscoelasticity of this interface and subsequent changes in ice cream volume during pressure or vacuum storage.

#### IV. CONCLUSIONS

Ice cream is one of the most complex food products, since it contains multiple phases (ice crystal dispersion, foam, emulsion, viscous unfrozen matrix, and potentially a weak gel system and a glass). Formation of the different phases is controlled during freezing, but the process of forming one phase generally influences the formation of the other phases. Thus the manufacture of ice cream requires careful control of both ingredient formulations and processing conditions. Since ice cream and related products are one of the few food products consumed in the semifrozen state, the freezing process is most important to ultimate smooth texture. As ice cream readily undergoes ice recrystallization, especially during periods of temperature fluctuation, precise control of frozen storage and distribution conditions also is critical for the preservation of optimal textural quality. For all these reasons, ice cream—type products present processing, storage, and distribution characteristics that are unique among the frozen foods.

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# 31

### **Effect of Freezing on Dough Ingredients**

#### María Cristina Añón

Universidad Nacional de La Plata, La Plata, Argentina

#### Alain Le Bail

ENITIAA-UMR GEPEA, Nantes, France

#### Alberto Edel Leon

Universidad Nacional de Córdoba, Córdoba, Argentina

Some modifications involving several constituents of bread formulation take place during preparation and baking of bread from frozen dough. This work analyzes the effect of freezing on the several ingredients, regardless of the processes and formulations used.

#### I. FLOURS

As in any baking process, flour quality is essential for obtaining a good product. In this case, the freezing process, transportation, possible temperature variations, and thawing are all factors demanding flours of better quality than those used for traditional baking.

In breadmaking, the proteins play a key role. After water addition, a cohesive dough is formed that is structured by gluten. Gluten proteins associated with lipids are responsible for dough cohesive and viscoelastic properties. These properties make the dough capable of retaining the gases produced by yeast action, leading, after baking, to a spongy product bearing elastic crumb. Processes that affect the proteins will also affect the quality obtained.

In the United States, flours recommended for breadmaking should have a protein content between 12 and 14% (Stauffer, 1993), though classical baking is carried out with flours containing 11% protein (Marston, 1978). European recommendations suggest flours with 12.5% protein with about 30% wet gluten (Brümmer, 1995). Nevertheless, the quality of these proteins may be as important as the amount, as shown by Inoue and Bushuk (1992). These authors studied the effect of freezing on baking performance with bread made of selected flours. These flours were similar in protein content (between 13.7 and 14.4%) but quite different in quality. Extensigraph results (maxima of resistance and extension) showed that dough strength and loaf volume decreased after freezing and thawing and during frozen storage. Some experiments were realized with constant yeast activity (at the same level as in nonfrozen doughs) and showed that loss of dough strength on freezing and thawing and during frozen storage was the main reason for the decline in

bread loaf volume. Inoue and Bushuk (1992) showed that protein content, in the range covered, appeared to be less important than protein quality.

During frozen dough processing, dough weakening occurs, which together with yeast damage is the main causes of the shortcomings of this methodology. This is evidenced by the production of bread loaves with lower volume, by the increase in fermentation times, and by alterations in textural properties (Dubois and Blockcolsky, 1986; Rasanen et al., 1997; Inoue and Bushuk, 1992; Wolt and D'Appolonia, 1984; and Neyreneuf and Van der Plaat, 1991).

The decrease of dough strength during freezing and the freezing—thawing cycles has been attributed to several factors. Ice crystal formation in nonfermented doughs stored for 24 weeks was found to cause rupture of the gluten network as observed in previous works by scanning electron microscopy (SEM) (Berglund et al., 1991).

Freezing process causes yeast death and the release of reducing substances; these effects were investigated. Some authors (Kline and Sughihara, 1968; Hsu et al., 1979) have proposed that owing to the reducing nature of these substances (mainly glutathione), disulfide bridges could be broken, thus leading to dough weakening. However, it has been suggested in other works that structural changes induced in frozen and thawed doughs are unrelated to the release of reducing substances (Varriano-Marston et al., 1980; Wolt and D'Appolonia, 1984; Autio and Sinda, 1992).

Recently, it was observed that, during dough storage at  $-18^{\circ}$ C, glutenin aggregates of molecular weight 129,100 and 88,700 experience depolymerization, which becomes more noticeable for longer storage times (Ribotta et al., 2001). This confirms that long-term frozen storage of doughs causes gluten depolymerization.

The release of reducing substances from dead yeasts added to the rupture of gluten network caused by ice crystals may explain the decrease of strength in frozen doughs, the loss of  $CO_2$  retention capacity, and the corresponding volume loss in bread loaves prepared with this technology.

Perron et al. (1999) have not found a clear correlation between the quality of breads obtained from frozen dough and protein content. However, it is known that the shortcoming of quality in this type of product is more noticeable when weak flours are used. In the U.S.A. it was reported that vital gluten supplementation improves product quality, lessening the difficulties mentioned above (Neyreneuf and Van der Plaat, 1991; Ribotta et al., 2001).

Starch is another important component of flour. This storage polysaccharide participates in the breadmaking process by absorbing water. For this reason, it is recommended for breadmaking from frozen dough that the flour used does not have more than 7% damaged starch (Marston, 1978), since excessively high levels of damaged starch increase the water absorption capacity of flours, creating problems during dough handling and fermentation (Pomeranz, 1988).

From the technological point of view, the role of starch is more important in bread firming. Although the nature of the physicochemical modifications that explain bread hardening is still widely discussed, a key role is assigned to starch in all hypotheses.

The mechanism causing bread firming has been studied for many years; its elucidation will allow the appropriate selection of methods to lessen this process. At first, the popular belief was that bread hardens with time only because of the moisture loss, but Boussinggault in 1852 stored hermetically packaged bread and also observed firming (Wilhoft, 1973).

A century later it was postulated that starch was responsible for hardening (Schoch and French, 1947). Later, several works contributed elements to relate bread firming with

starch retrogradation (Kim and D'Appolonia, 1977a; Kim and D'Appolonia, 1977b; Eliasson, 1985; Inagaki and Seib, 1992, León et al., 1997a). However, other results were in conflict with the hypothesis explaining bread firming as a consequence of amylopectin retrogradation: (a) the rate of bread hardening is linear up to day 5 while amylopectin retrogradation rate increases linearly up to day 3 (Ghiasi et al., 1984). (b) Breads with low moisture content harden faster, while starch retrogradation rate is unaltered (Rogers et al., 1988); (c) breads with  $\alpha$ -amylase as additive harden more slowly, but they show increased crystallinity as studied by x-ray (Dragsdorf and Varriano-Marston, 1980).

Upon these results Martin et al. (1991) have proposed a bread firming mechanism based on the increase in interactions between starch molecules and gluten proteins. However, later works have shown that gluten addition in model systems did not affect firming rate (León et al., 1997b; Durán et al., 2001).

León et al. (1997a) have developed a new technique for studying the changes occurring during bread storage, which is based on direct observation of the transformations in progress. To this end, bread dough is baked in a differential scanning calorimeter capsule, following the temperature profile at the crumb center, to test samples by new calorimetric runs after different storage times. By applying this methodology to frozen dough, greater retrogradation rates were found in samples kept frozen for more than 30 days compared with control samples.

By deepening this research, it was found in frozen samples stored for 60 days and then baked in the DSC capsules that amylopectin retrogradation was faster than in fresh doughs. When stored at both 4 and 20°C, in the former temperature, the effect of freezing was more noticeable (Table 1) (Ribotta et al., 2003a).

#### II. YEASTS

Freezing causes stress to microorganisms. Five major factors may affect the cell during freezing (Mazur, 1976); (a) low temperature, (b) extracellular ice formation, (c) intracellular ice formation, (d) concentration of the extracellular solute, and (e) concentration of intracellular ice. (d) and (e) can be due to ice formation (intra- or extracellular) or to water/solute diffusion through the cell membrane. Factors (a) and (b)

Table 1	Effect of Storage at Diffe	rent Temperatures on A	Amylopectin l	Retrogradation (	$(\Delta H_{\rm R})$
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	Stored at 4°C		Stored at 20°C	
Storage time (days)	Bread	Bread obtained from frozen doughs	Bread	Bread obtained from frozen doughs
0	$-0.07 \pm 0.01$	$-0.07 \pm 0.01$	$-0.07 \pm 0.01$	$-0.07 \pm 0.01$
24	$-1.13 \pm 0.06$	$-1.92 \pm 0.13$	$-0.94 \pm 0.09$	$-1.11 \pm 0.19$
48	$-1.76 \pm 0.59$	$-2.89 \pm 0.07$	$-1.30 \pm 0.06$	$-1.32 \pm 0.18$
96	$-2.22 \pm 0.29$	$-3.44 \pm 0.17$	$-1.82 \pm 0.15$	$-1.92 \pm 0.07$
144	$-3.01 \pm 0.06$	$-3.46 \pm 0.04$	$-2.03 \pm 0.09$	$-2.19 \pm 0.0$
168	$-3.12 \pm 0.24$	$-3.68 \pm 0.03$	$-2.18 \pm 0.06$	$-2.16 \pm 0.21$

cannot cause per se cell damage (Mazur, 1976). Most microorganisms can support cell dehydration. Factors (c) and (d) are most likely to be the major phenomena involved in cell damage. Intracellular freezing is very difficult to achieve in most conventional industrial freezing processes. Freezing rates in excess of 10°C/min to 100°C/min can produce intracellular ice formation for yeast and bacteria, respectively (Mazur, 1976). The lower the size of the cell, the higher the freezing rate should be; this is because of the cell volume and the thermodynamics associated with ice crystallization. It has been demonstrated that a critical ice nucleus radius exists, below which the nuclei are not stable and cannot exist or grow (Fennema et al., 1973). The conventional freezing process as the one used to freeze bread dough is limited in term of freezing rate. Yeast is a quite resistant microorganism, but the activity of yeast is affected by freezing.

Higher yeast proportions are thus recommended in frozen dough formulations to compensate for the activity loss due to freezing per se and for the storage periods, leading to lower gas production capacity (Lorenz and Kulp, 1995). This is provoked by the changes induced during freezing, which can cause yeast death. Cells exposed to temperatures below 0°C are damaged in different ways depending on the temperatures reached and the cooling rate.

Although the freezing point of the cytoplasmic content of cells is about  $-1^{\circ}$ C, it can remain unfrozen even in the presence of ice crystals in the external medium (Mazur, 1965). The higher water vapor pressure in the cell interior compared with that of the external ice causes water loss. This dehydration equilibrates vapor pressures on both sides of the membrane, leading to solute concentration in the cytoplasm (Mazur, 1970).

It has been suggested that the optimum cooling rate for destroying as few yeast cells as possible is 7°C/min. For rates below the optimum, the proportion of deaths increases by the "solution effect" (increase in concentration), while rates above the recommended values increase the damage by intracellular ice formation (Mazur, 1970). Besides, ice crystals' recrystallization leads to cell death by causing damage inside the cell and on the plasmatic membrane.

The solution effect is explained by four stages occurring during freezing: (a) water is transferred toward the ice, (b) solutes become concentrated, (c) cell volume decreases, and (d) some solutes precipitate (Casey and Foy, 1995).

The results of Mazur (1970) were obtained with cell suspension and cannot be directly applied to the case of frozen dough. When incorporated in the bread dough, yeast does not behave the same way as yeast alone. The relationship between freezing and yeast activity becomes more complex. The proportion of dead cells caused by freezing is higher in yeast incorporated into the dough than in freezing yeasts (Lorenz and Kulp, 1995; Ribotta et al., 2003a) (Table 2).

This phenomenon can be explained by considering that active cells (as those found in the dough) have their plasma membranes thinner than in dormant cells and therefore less

Table 2 Dead Yeasts Percentage During Freezing Dough and Yeasts for Several Storage Times

Time (days)	0	40	60	90
Frozen dough	2.7	7.5	20.2	27.7
Frozen yeast	1.4	1.7	2.5	5.2

resistant to damage. Besides, molecules produced by fermentation, such as ethanol, aceticacid, and lactic acid, concentrate in the unfrozen region of the aqueous phase. This concentrated solution of organic substances can produce autolysis of yeast cells, as it is known that active cells are more sensitive to this autolytic action (Hsu et al., 1979).

The decrease in yeast yield exceeds cell death, since CO<sub>2</sub> production losses of 13.2% are found in ready frozen dough; this loss increases to 37.7% at 45 days and to 52.4% at 60 days of frozen storage. Therefore freezing causes yeast death and impairs CO<sub>2</sub> production capacity of the surviving yeast. By adding frozen yeast extract to bread formulation, it was observed that the longer the yeast remained in the frozen state before extraction, the lower the specific volume of bread obtained with that extract (Ribotta et al., 2003b).

The dissimilar sensitivity of yeasts of different origins is related to lipid composition, mainly the sterol/phospholipid ratio, which affects plasma membrane fluidity (Murakami et al., 1996). In recent years, work has been carried out to obtain yeast strains of improved resistance to freezing, in order to use them in breadmaking via frozen dough (Nakatomi et al., 1885; Uno et al., 1986; Takano et al., 1990; Baguena et al., 1991; Van Dijck et al., 2000).

Other substances were used to protect yeasts, such as trehalose (Coutinho et al., 1988; Meric et al., 1995). This carbohydrate is known to be an efficient protective agent to preserve membrane integrity and intracellular structure in a wide range of physiological and room conditions (Van Laere, 1989). Meric et al. (1995) showed that a 5% minimum trehalose content was necessary to achieve a significant improvement of yeast resistance to freezing. No real benefit was observed above this limit. For this reason, in new yeast strains, efforts are directed to make trehalose synthesis more active and to lessen the effect of catabolic routes (Casey and Foy, 1995).

#### III. OXIDIZING AGENTS

The role of oxidizing agents is essential in breadmaking because they increase gluten strength and allow breads of higher volume to be obtained. In breadmaking via frozen dough, oxidant addition is particularly necessary; owing to protein matrix weakening caused by the mechanical action of ice and by the effect of reducing substances released by yeasts.

In the United States, flour for breadmaking via frozen dough is usually given 45 ppm potassium bromide combined with 100 ppm ascorbic acid (Stauffer, 1993). After potassium bromide prohibition as a breadmaking additive in almost all the world, azodicarbonamide was proposed as substitute, to be used in combination with ascorbic acid and enzymes such as lipoxygenase, or else enzymatic complexes contained in active soy flours.

The comparative analysis of potassium bromide and ascorbic acid actions showed that both improve product quality and that the quality of bread given ascorbic acid is higher than that added with potassium bromide (Abd El-Hady et el., 1999).

Azodicarbonamide is a "quick action" oxidant, very sensitive to glutathione and to other substances released on yeast death (De Stefanis, 1995). Thus it is best used, according to recommendations, when combined with benzoyl peroxide (De Stefanis, 1994). Concerning fermentation time, (Dubois and Blockolsky (1976)) showed that potassium bromide, used in low concentrations, shortens fermentation time compared to that obtained with ascorbic acid.

If potassium bromide is used in high concentrations, it is found that from 20 weeks of frozen storage on, fermentation times become longer than those obtained with ascorbic acid, potassium iodide, and especially than that found when using azodicarbonamide (De Stefanis, 1995).

#### IV. ADDITIVES

Emulsifiers are increasingly used in those bakery products that include fat in their formulations, since they improve dough stability and the retention capacity of the gases produced by fermentation; they also improve loaf volume and crumb freshness with longer shelf-life.

For frozen doughs, effective additives have been proposed to improve product quality such as SSL and DATEM (Wolt and D'Appolonia, 1984). By adding SSL, freezing-induced quality losses can be alleviated (Adb El-Hady et al., 1999). DATEM addition significantly increased volume, shape ratio, and bread quality (Sahlström et al., 1999).

Glycerol used at 0.75–1.5% (on a flour basis) causes little effect on the volume obtained via frozen doughs, though crumb structure is improved. In turn, xanthan gum addition does not improve volume and reduces crumb quality (Dubois and Blockolski, 1986).

In order to have a flour bearing the required "strength," a 2% addition of gluten has been proposed as the optimum level to improve bread quality, gas retention capacity, and fermentation time (Wang and Ponte, 1994). Apart from improving protein quality, a cryoprotectant effect on yeast is obtained, since on increasing glass transition temperature the free water proportion is decreased (Levine and Slade, 1986). Other additives capable of reducing, at least partially, the shortcomings caused by dough freezing have been described. Among them, we find egg yolk and sugar esters (Hosomi et al., 1992) and the addition of vegetable fats to form water/oil emulsions at different proportions (Inoue et al., 1995).

A summary of the principal additives and their effects on the characteristics of frozen bread is shown in Table 3.

#### V. CONCLUSIONS

Throughout frozen dough processing, dough weakening occurs that together with yeast damage is the main cause of the shortcomings of this technology. Increases in fermentation times, the production of bread loaves with lower volume, and alterations in textural properties are detected. The success of the expanding use of frozen dough for the production of bread and bakery products depends of the resolution of these problems.

The fact that the freezing process affects the viability of yeast cells and the production of CO<sub>2</sub> opens an important technological and research field. We need to find new strains of yeasts and search for additives and process variables that will help the cryoresistence of the microorganisms.

Another important phenomenon that deserves attention is protein depolymerization during frozen storage, which may partially reduce the need for different additives.

In the production of bread from frozen dough, complex formulations must be used to reduce the harmful effects of freezing. These formulations include lipids, high

Table 3 Principal Additives Used in the Formulation of Frozen Dough

Additive	Effect	Reference
Gluten 2%	Increase dough strength	Wang and Ponte, 1994
SSL 0.5%	Decrease the freezing effect on bread volume	Abd El-Hady et al., 1999
DATEM 0.6%	Increase bread volume	Sahlström et al., 1999
Mix of sucrose, fatty acid ester, DATEM, fatty acid monoglycerides, and sugar	Avoid the harmful effects of freezing on the quality of baking products	Nakamura et al., 1996
Mix of gums, tensioactive agents, and forming film proteins	Avoid the harmful effects of freezing on the quality of baking products	Larson et al., 1983
Mix of gluten, emulsifier, and polymeric substances	Inhibit the deterioration of dough, eliminating the damage produced by big ice crystals	Yamaguchi and Watanabe, 1987
Enzymes that produce maltotrioses and glucose oxidase and/or hemicelulase	Prevent volume reduction due to freezing	Tanaka et al., 1997
Glycerol 0.75–1.0%	Improve the structure of the crumb	Dubois and Blockolski, 1986
Ethanol-water 5–20%	Decrease the water melting point and diminish the thawing time previous to fermentation	Lidstrom and Slade, 1987
Sucroglycerides	Protect the yeast against the harmful effect of freezing on baking products	Le Duff, 1987a,b
Lecithin 0.2–0.3%	Flour strength	Grandvoinnet et al., 1986
Alfa-amylase 0.05–0.1%	Produce fermentable carbohydrates	Larsen, 1991
Skim milk—whey protein 2.2%	Provide humidity in baking products, avoiding the use of gluten that alters the organoleptic properties	Seneau, 1989

percentages of yeast and oxidizing agents, and mixes of different oxidizing agents, surfactants, or emulsifiers that improve the bread matrix, gluten, soy flour, sugars, and enzymes. For this reason, the freezing process is more profitable for bakery products with a high added value.

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### Microwavable Frozen Food or Meals

Kit L. Yam

Rutgers University, New Brunswick, New Jersey, U.S.A.

Christopher C. Lai

Pacteco Inc., Kalamazoo, Michigan, U.S.A.

#### I. INTRODUCTION

The microwave oven was first developed during the 1950s and became a popular household appliance when its penetration level soared during the 1980s—currently, more than 90% of U.S. households own at least one microwave oven. The major driving forces for the microwave oven are changing lifestyles and the development of microwavable food products.

The changing lifestyles in recent years (two-income families, single parents, schoolage children home alone) are increasingly putting a premium on convenience and quick preparation of food. The microwave oven captures this opportunity by providing a convenient means for the consumer to cook or reheat food quickly and easily. At the same time, the food industry has also developed new products or reformulated existing products for the microwave oven. Microwavable food products are now ubiquitous in the supermarket. In particular, microwavable frozen meals are a major category, since they provide a convenient solution to people who do not have time to prepare their own meals. The packaging industry has also contributed to developing new technologies and packages that are compatible to microwave heating. The consumer can quickly microwave a frozen meal in a container and enjoy the meal from the same container.

While microwavable food products have made significant inroads in the past two decades, there still remain many challenges for the developer and manufacturer. Besides convenience, other factors such as taste and texture are also important to the consumer. Quite often, the consumer perceives food heated by the microwave oven as not tasting as good as that heated by the conventional oven. This is because the heating mechanisms of food in the microwave oven and conventional oven are indeed quite different. To develop successful microwavable food products, the developer must have a good understanding of the microwave heating of food and give careful consideration of the package design and consumer expectation.

## II. BASICS OF MICROWAVE HEATING OF FOOD

Microwave heating of food is a complex process that requires a good understanding of several relevant disciplines: electromagnetism, food engineering, food chemistry, food packaging, and food microbiology. It is beyond the scope of this chapter to provide detailed descriptions of the many aspects of this complex process. Instead, this chapter is aimed at acquainting the reader with the basic working knowledge most relevant to the microwave heating of frozen foods. More general information can be found from references in the literature (1–3).

## A. Microwaves

Microwaves are short electromagnetic waves located in the portion of the electromagnetic spectrum between radio waves and visible light. The energy is delivered in the form of propagating sine waves with an electric field and a magnetic field orthogonal to each other. Microwaves are relatively harmless to humans because they are a form of nonionizing radiation, unlike the much more powerful ionizing radiation (such as x-rays or gamma rays) that can damage the cells of living tissue. Microwaves are used in daily applications such as cooking, radar detection, and telecommunications.

Most microwave ovens for food applications operate at two frequencies. The household microwave oven operates at 2450 MHz ( $2.45 \times 10^9$  cycles per second), and the industrial microwave oven operates at 915 MHz ( $9.15 \times 10^8$  cycles per second). The wavelengths associated with these frequencies are 0.122 and 0.382 m, respectively, when the microwaves are assumed to travel at the speed of light ( $3 \times 10^8$  m/s). Microwaves travel at approximately the speed of light in air, but they travel at a lower speed inside a food material. The relationship between frequency and wavelength is expressed by the equation  $v = f\lambda$ , where v is the velocity (m/s), f is the frequency (Hz), and  $\lambda$  is the wavelength (m) of the electromagnetic wave.

There are three possible modes of interaction when microwaves impinge upon a material: absorption of microwaves by the material, reflection of microwaves by the material, and transmission of microwaves through the material. The material may be a food or a packaging material. The food must absorb a portion of the microwave energy in order for heating to occur. Most foods do not reflect microwaves, and thus all the remaining unabsorbed microwave energy is transmitted. Some packaging materials, such as susceptors, absorb microwave energy and become hot. Metals, such as aluminum foils, reflect microwaves. Paper, plastics, and glass are transparent to microwaves. To optimize the microwave heating of food, it is necessary to consider the reflection, absorption, and transmission of microwaves by the food and the package.

In the microwave oven, microwaves are generated by an electronic vacuum tube known as a magnetron. The microwaves then travel through a hollow metal tube called a waveguide to the oven cavity. To improve the heating uniformity, the microwave oven is often equipped with a stirrer or a turntable. The stirrer is a fanlike set of spinning metal blades used to scatter the microwaves and disperse them evenly within the oven. The turntable rotates the food during the microwave process. The history, features, standardization, and safety matters relating to the microwave oven are discussed by Decareau (1).

#### B. Microwave/heat Conversion

Microwave energy is not heat energy. In order for microwaves to heat food, they must first be converted to heat. There are two mechanisms by which this energy conversion can occur: dipole rotation and ionic polarization. The two mechanisms are quite similar, except the first involves mobile dipoles while the second involves mobile ions. Both dipoles and ions interact only with the electric field, not the magnetic field.

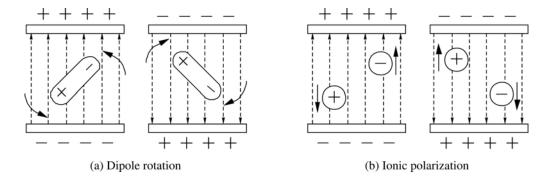
Figure 1a illustrates the dipole rotation mechanism of a polar molecule. In the presence of an electrical field, the polar molecule behaves like a microscopic magnet, which attempts to align with the field by rotating around its axis. As the polarity of the electric field changes, the direction of rotation also changes. The molecule thus absorbs microwave energy by rotating back and forth billions of times at the frequency of microwaves. Since the molecule is often bound to other molecules, the rotating action also causes it to rub against those other molecules. The rubbing action disrupts the bonds between the molecules, which in turn causes friction and heat dissipation.

The water molecule is the most abundant polar molecule in food. The water molecules in liquid water are quite mobile, and they readily absorb microwave energy and dissipate it as heat through dipolar rotation. On the other hand, the water molecules in ice are much less mobile owing to the confined crystal structure, and they do not absorb microwaves well. The distribution of moisture and the state of water (liquid or ice) are often two critical factors that determine the behavior of the microwave heating of foods.

Figure 1b illustrates the ionic polarization of a positive ion and a negative ion in solution. In the presence of an electric field, the ions move in the direction of the field. As the polarity of the electric field changes, the ions move in the opposite direction. The ions absorb microwave energy by oscillating at microwave frequencies. The oscillating action in turn causes heat dissipation through friction. The common ions in food are those from salts such as sodium chloride. Since ions are less abundant than water molecules in most foods, ionic polarization often plays a less important role than dipole rotation.

## C. Dielectric Properties

While dipole rotation and ionic polarization provide a qualitative understanding of microwave/heat conversion mechanisms, the dielectric properties provide a quantitative



**Figure 1** Microwave/heat conversion mechanisms. The dashed lines denote the alternating electric field at the frequency of microwaves. (a) A dipole rotates back and forth. At high frequencies (such as 2450 MHz), there is not sufficient time for the dipole to rotate 180°, and thus the actual rotation angle is much smaller. (b) A positive ion and a negative ion oscillate in an alternating electric field.

characterization of the interactions between microwave electromagnetic energy and food. The dielectric properties, along with thermal and other physical properties, determine the heating behavior of the food in the microwave oven.

An important dielectric property is dielectric loss factor ( $\varepsilon''$ ), which indicates the ability of the food to dissipate electrical energy. The term loss refers to the loss of energy in the form of heat by the food. It is useful to remember that a material with a high  $\varepsilon''$  value (also known as a lossy material) heats well, while a material with a low  $\varepsilon''$  value heats poorly in the microwave oven. The dielectric loss factor is related to two other dielectric properties by the equation

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'} \tag{1}$$

where tan  $\delta$  is loss tangent and the  $\varepsilon'$  is a dielectric constant.

The dielectric properties ( $\varepsilon'$  and  $\varepsilon''$ ) are functions of frequency, temperature, moisture content, and salt content. Values of dielectric properties for foods and other materials can be found in the literature (4–6). Examples of  $\varepsilon''$  and  $\varepsilon'$  values at 2450 MHz are shown in Table 1. Although the literature values can be used as guidelines, actual measurements are often required because of the variability of composition of the materials.

The dielectric properties provide a quick indication of how well a material heats in the microwave oven. For example, the  $\varepsilon''$  value of water (12.48) at 25°C is several orders of magnitude higher than that of ice (0.0029) at -12°C. This means that water heats far better than ice in the microwave oven. Ice is almost transparent to microwaves because its molecules are tightly bound and do not rotate easily through the mechanism of dipolar

**Table 1** Dielectric Constant ( $\varepsilon'$ ), Dielectric Loss Factor ( $\varepsilon''$ ), and Penetration Depth ( $D_p$ ) of Various Foods at 2450 MHz

	$\varepsilon'$	$\varepsilon''$	D <sub>p</sub> (cm)	
$\overline{\text{Ice } (-12^{\circ}\text{C})}$	3.2	0.0029	1203	
Water (1.5°C)	80.5	25.0	0.71	
Water (25°C)	78	12.48	1.38	
Water (75°C)	60.5	39.93	0.40	
0.1 M NaCl (25°C)	75.5	18.1	0.94	
Fat and oil (average)	2.5	0.15	20.6	
Raw beef (-15°C)	5.0	0.75	5.83	
Raw beef (25°C)	40	12	1.04	
Roast beef (23°C)	28	5.6	1.85	
Boiled Potatoes (-15°C)	4.5	0.9	4.6	
Boiled Potatoes (23°C)	38	11.4	1.07	
Boiled spinach (-15°C)	13	6.5	1.11	
Boiled spinach (23°C)	34	27.2	0.45	
Polyethylene	2.3	0.003	986	
Paper	2.7	0.15	21.4	
Metal	$\infty$	0	0	
Free space	1	0	$\infty$	

rotation. The dramatic increase in  $\varepsilon''$  value is also observed, when ice changes to water, during the thawing of frozen foods including beef, potato, and spinach (Table 1). Plastics and paper have low  $\varepsilon''$  values because they are almost transparent to microwaves.

## D. Penetration Depth

The speed of microwave heating is due to the deep penetration of microwaves into the food; the dielectric properties can be used to determine the extent of penetration. When microwaves strike a food surface, they arrive with some initial power level. As microwaves penetrate the food, their power is attenuated, since some of their energy is absorbed by the food. The term penetration depth  $(D_p)$  is defined as the depth at which the microwave power level is reduced to 36.8% (or 1/e) of its initial value, which can be estimated using the equation

$$D_{\rm p} = \frac{\lambda_0}{2\pi\sqrt{2\varepsilon'}} \left[ \left( 1 + \tan^2 \delta \right)^{1/2} - 1 \right]^{-1/2} \tag{2}$$

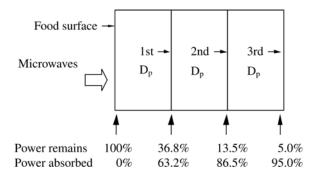
where  $\lambda_0$  is wavelength in free space. At 2450 MHz,  $\lambda_0 = 12.24$  cm, and

$$D_{\rm p} = 1.38 \left\{ \varepsilon' \left[ \left( 1 + \tan^2 \delta \right)^{1/2} - 1 \right] \right\}^{-1/2}$$
 (3)

where  $D_p$  is in cm. The penetration depth is a visual term that describes how well a food absorbs microwaves: the shorter is the penetration depth, the more the food absorbs microwaves.

The meaning of penetration depth is further illustrated in Fig. 2. At the first  $D_{\rm p}$ , 36.8% of the initial power remains, while 63.2% of the power is absorbed. At the second  $D_{\rm p}$ ,  $(0.368)^2 = 13.5\%$  remains, and 86.5% is absorbed. At the third  $D_{\rm p}$ ,  $(0.368)^3 = 5.0\%$  remains and 95% is absorbed. The penetration depth depends on the composition of the material, the frequency of microwaves, and the temperature.

Typical values of  $D_{\rm p}$  for various materials at 2450 MHz are also shown in Table 1. As mentioned earlier, liquid water absorbs microwaves far better than ice. The  $D_{\rm p}$  of water at 25°C is 1.38 cm, but the  $D_{\rm p}$  of ice at -12°C is 1203 cm! Frozen foods have longer penetration depths than unfrozen foods. For example, the  $D_{\rm p}$  values for frozen beef and unfrozen beef are 5.83 cm and 1.04 cm, respectively.



**Figure 2** Power levels at various penetration depths.

## E. Mathematical Equations and Models

Besides Eq. (2), other simple equations can also provide researchers and food product developers with a better understanding of the microwave heating process. For example, microwave power absorption can be estimated using the equation

$$P = k\varepsilon'' f E^2 \tag{4}$$

where P is power absorption (watts/cm<sup>3</sup>), k is constant (5.56 ×  $10^{-13}$  farads/cm),  $\varepsilon''$  is a dielectric loss factor of food (dimensionless), f is the frequency of the microwaves (Hz), and E is electric field strength of the microwaves (volts/cm). The power absorption is directly proportional to the dielectric loss factor, indicating that a lossy material (which has a high  $\varepsilon''$  value) is also a good absorber of microwave energy.

The rate of the temperature increase of the food can be estimated using the equation

$$\frac{dT}{dt} = \frac{k\varepsilon'' f E^2}{\rho C_{\rm p}} \tag{5}$$

where T is the average temperature of the food (°C), t is time (s),  $\rho$  is the density of the food ( $g \cdot cm^{-3}$ ), and  $C_p$  is the specific heat of the food ( $Jg^{-1} \circ C^{-1}$ ). Equation (5) is an energy balance equation, which assumes that the microwave energy absorbed is balanced by the heat gain of the food. Note that this is a relatively simple equation, and it does not consider the fact that temperature is not evenly distributed within the food.

Mathematical models based on heat and mass transfer principles are also available to provide more sophisticated information during the microwave heating of food (7,8). A typical model consists of a set of partial differential equations with the proper initial and boundary conditions. The models can be used to predict the temperature and moisture distribution histories of foods during microwave heating. To use the models, values for dielectric properties, thermal properties, density, electrical field strength, and product dimensions are required. Models for frozen food are more complicated than those for unfrozen food, because the microwave heating behavior changes greatly from the frozen state to the unfrozen state.

The models can simulate what-if scenarios and thus can help to minimize the number of experiments and shorten the product development time. However, the models are limited mostly to the predictions of temperature and moisture content, and they do not deal with other important factors such as taste and texture. Most models are also limited to foods that are homogenous and have regular shapes.

#### III. CHALLENGES IN MICROWAVE HEATING OF FROZEN FOOD

While microwave heating offers the benefits of speed cooking and convenience, it also presents many technical challenges to the food scientist or technologist. Those challenges arise from the need to deal with the many variables relating to the food, package, and microwave oven. For the food, there are the variables of food composition, shape, size, specific heat, density, dielectric properties, and thermal conductivity. For the package, there are the variables of shape, size, and properties of packaging material. For the microwave oven, there are variables relating to the design of the oven. A related and more important challenge is to solve the problems of the consumer. From the consumer's point

of view, the most noticeable problems are those associated with nonuniform heating, lack of browning and crisping, and variation in microwave ovens.

## A. Nonuniform Heating

Nonuniform heating is a major problem in microwave heating. The problem is especially noticeable for frozen food. It is not uncommon for a frozen food heated in a microwave oven to boil around the edges while the center remains frozen. The problem is caused by the differences in microwave energy absorption of liquid water and ice.

In frozen foods, the water molecules on the surface are relatively free to move compared to the water molecules inside the food. When a frozen food is microwaved, heating begins at the surface where the water molecules are more ready to absorb microwave energy. This causes the adjacent ice crystals to melt and the surface temperature to rise, while the inside temperature is still little affected. As more liquid water is available, the heating of the surface becomes more rapid. This can lead to runaway heating, in which the heating is excessive at the surface while the inside is still frozen. To minimize runaway heating during thawing, microwave energy should be delivered at a slow rate, which allows more time for heat to spread from the surface to the inside.

An irregular shape of a food can also cause nonuniform heating. The thin parts tend to overcook, while the thick parts tend to undercook. This situation also occurs in conventional cooking but is less pronounced because the cooking is slower. Another cause of nonuniform heating is that different foods have different dielectric and thermal properties. When a microwave meal consists of two or more items, it is possible that the items heat at different rates. For example, when microwave heating a frozen meal consisting of meat and vegetable, the vegetable often becomes overheated and dried out before the meat reaches the serving temperature.

## B. Lack of Browning and Crisping

Another problem is that, unlike the conventional oven, the microwave oven is not able to produce foods that are brown and crisp. This is because the heating mechanisms of the conventional oven and the microwave oven are quite different.

In the conventional oven, the food is heated by hot air in the oven, and if the heating element is not shielded, the food is also heated by radiated heat. Heating is concentrated on the food surface by means of heat convection and radiation. The inside of the food is also heated, at a slow rate, by means of heat conduction. The heating causes the moisture on the food surface to evaporate rapidly, and later, browning and crisping begin. Although the moisture inside the food tends to migrate to the surface, the rate is not sufficiently fast to prevent browning and crisping. As a result, the food surface becomes brown and crispy while its inside remains moist and soft.

In the microwave oven, there is no hot air, and heating is mostly due to the interaction between microwaves and water. Microwave heating is not concentrated on the food surface, but it is distributed within the food depending on the penetration depth. The heating on the food surface is no longer sufficiently intense to cause browning and crisping. Unless the food is microwaved for a long time to remove all or most of the water in the food (which is not desirable because the food quality may no longer be acceptable), browning and crisping either do not occur at all or are inadequate.

Browning formulations have been developed for various meat and dough products (1). Commercial steak sauces, barbecue sauces, soy sauces and the like are brushed on meat before microwave heating. Reusable browning dishes are also available for browning food surfaces in the microwave oven. Most of the commercial browning dishes are made of glass-ceramic substrate with tin oxide coating on the underside. The packaging industry has also developed a disposable browning and crisping material, known as susceptor, discussed later in this chapter.

## C. Variation in Microwave Ovens

Yet another problem is the large variation of performance in different microwave ovens. Microwave ovens are available in different powers, different oven cavity sizes, and with or without a turntable and with or without a stirrer (to distribute microwaves more evenly in the oven). Consequently, different microwave ovens may produce greatly different results, even if the same cooking instructions are used. To accommodate the differences, the food manufacturer can only place vague microwave heating instructions on their packages. For example, a package may contain vague instructions such as "heat between 4 to 8 minutes, depending on the microwave oven."

## D. Meeting the Challenges

There is no easy solution to deal with the complex process of microwave heating. In developing a microwavable food product, the scientist or technologist has to rely on the somewhat useful but incomplete scientific knowledge described in the previous sections, as well as trial-and-error or empirical methods.

There are three approaches to deal with the challenges. The first is the food chemist's approach, in which food ingredients are modified and browning formulations are added to make the food more microwavable. The second is the packaging engineer's approach, in which the package is modified to enhance the performance of microwave heating. The third is the microwave engineer's approach, in which new and useful features are added to the microwave oven. Ideally, these approaches should be integrated into a system to deliver the highest quality of microwavable foods to the consumer.

Many microwavable food products have failed in the past because of lack of performance or high cost. Good technical and marketing tools are essential for developing better tasting microwavable products, without increasing the cost or decreasing the effectiveness of cooking. Although the food manufacturer and the packaging supplier have been working together to develop microwavable products, there has been relatively little collaboration between them and the oven manufacturer. There is a need to have all parties (including also academia) to work more closely together to bring about innovations that can deliver better microwavable products to the consumer.

#### IV. MICROWAVABLE PACKAGING

The primary functions of the package are to contain, protect, and sell the product. A general discussion on the packaging of frozen foods is presented in Chapter 6. If the package is used to hold the food during microwave heating (as is the case for many microwavable frozen meals), the interactions between the microwave and the package must also be considered. Since the package can transmit, reflect, or absorb microwaves, it

can also greatly influence the microwave heating behavior. The package may act passively by simply transmitting microwaves. The package may also act actively by reflecting and absorbing microwaves so that the power distribution of microwaves and the surface temperature of the package are modified. To optimize microwave heating, it is necessary properly to balance these three microwave/package interactions (transmission, reflection, and absorption) to optimize the heating of the food. Microwavable packaging materials may be classified into microwave transparent materials, microwave reflective materials, and microwave absorbent materials.

## A. Microwave Transparent Materials

A microwavable package must be wholly or partly transparent to microwaves. The most common microwave transparent materials are paper and plastics. Although glass is also transparent to microwaves, it is seldom used to package frozen food.

Plastic-coated paperboard trays are popular for microwavable frozen meals, mainly because of their low cost. The trays combine the rigidity of the paperboard and the chemical resistance of the plastic. The inside of the trays is either extrusion coated with a resin or adhesive laminated with a plastic film. For microwave-only applications, plastics such as low-density polyethylene (LDPE), high-density polyethylene (HDPE), and polypropylene (PP) are used. For dual oven applications (i.e., usable in both microwave and conventional ovens), polyethylene terephthalate (PET) is used because of its high temperature stability (up to about 200°C).

Molded pulp trays are another common paper product. The containers are dual ovenable and can be molded into several compartments. They are stronger and can carry more load than the paperboard containers.

Thermoformed plastic trays are also common heating containers for microwavable frozen foods. LDPE trays are suitable for light microwave heating because the trays tend to distort at temperatures as low as 75°C. PP trays have a distortion temperature of about 110°C. Homopolymer PP trays are brittle at low temperatures and can crack during distribution and handling at freezer temperatures. Copolymer PP trays have somewhat improved low-temperature durability. Crystallized PET (CPET) trays are the most widely used plastic trays for microwavable frozen meals. The CPET trays are functional in the temperature range from -40 to 220°C. Thus the trays can withstand not only the low temperatures encountered in distribution and handling but also the temperatures in conventional oven (i.e., the trays are dual ovenable).

#### B. Microwave Reflective Materials

Aluminum foil, aluminum/plastic laminate, and aluminum/plastic/paperboard laminate are the most common microwave reflective materials. Since these materials do not allow the transmission of microwaves, they are also known as microwave shielding materials.

Aluminum is often used to shield microwaves selectively from certain areas of a food (Fig. 3). For example, a multicomponent meal may consist of food items that heat at different rates in the microwave oven. The more microwave sensitive food item(s) can be shielded so that the entire meal can be heated more evenly.

Aluminum is also used as an electromagnetic field modifier to redirect microwave energy so as to optimize the heating performance (10). Aluminum can intensify the microwave energy locally or redirect it to places in the package that otherwise would



Figure 3 Trays with selectively shielded areas. (Courtesy of Graphic Packaging Inc.)

receive relatively little direct microwave exposure. This approach has been used to redirect microwave energy from the edges to the center for frozen food products such as lasagna.

When aluminum foils are used in the microwave oven, precautions are necessary to prevent arcing, which can occur between foil packages and the oven walls, between two packages, across tears, wrinkles, and so on. Arcing can be prevented by following several simple design rules (11). For example, any foil components should be kept back from the edge of the package to avoid arcing with the oven walls. In additional to following these rules, it is also necessary to thoroughly test the package/product to ensure that the package is safe to use.

#### C. Microwave Absorbent Materials

Microwave absorbent materials used for food packaging are commonly known as susceptors. The major purpose of susceptors is to generate surface heating to mimic the browning and crisping ability of the conventional oven. Although many types of susceptors have been invented (12), the only commercially available type is the metallized film susceptor (Fig. 4). This type of susceptor consists of a metallized polyethylene terephthalate film laminated to a thin paperboard. The metal layer is a very thin (less than



Figure 4 Metallized film susceptors. (Courtesy of Graphic Packaging Inc.)

100 angstroms) discontinuous layer of aluminum, which is responsible for generating localized resistance heating when exposed to microwaves. The heating can cause the susceptor to reach surface temperatures over 200°C within seconds.

Susceptors have been used for products such as frozen pizza, frozen French fries, frozen waffles, frozen hot pies, and popcorn. Susceptors are available in the forms of flat pads, sleeves, and pouches. The flat pads are suitable for products (such as pizza) that require heating only on one surface. The sleeves and pouches are suitable for heating on multiple surfaces (9). Susceptors are also available in various patterns, in which portions of the metallized layer are deactivated (10). The patterns are designed to provide more control of heating. A company uses a printed checkerboard pattern to generate various levels of heating based on the size of the check.

There is public concern about the migration of mobile compounds from the susceptor to the food, because the susceptor can reach high temperatures. The FDA has issued voluntary guidelines regarding the safe use of the susceptor for packaging.

#### V. ADVANCED OVENS

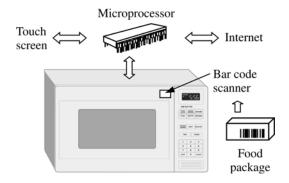
Although the oven manufacturers have made many improvements to the microwave oven, especially during the last two decades, it has encountered the challenges described earlier. Recently, the oven manufacturers have responded to those challenges by introducing more advanced ovens, such as high-speed ovens and intelligent ovens. These advanced ovens are superior to the microwave oven, and they can provide better heating for frozen food products.

#### A. High-Speed Ovens

High-speed ovens are essentially multimode ovens equipped with advanced technologies. The move toward using multimode heating instead of single-mode heating to deliver higher speed and food quality is a technically sound one, because any single-mode heating (microwave or convective heat) has too many inherent limitations. The current commercial high-speed ovens differ from one another in terms of hardware and software.

In hardware, the major difference is in the heating sources: microwaves, convective heat, hot-air impingement, and halogen light. Although it is possible to use more than two heating sources, most ovens are limited to only two heating modes because of cost and power consumption. High-speed ovens typically use microwaves to provide speed cooking along with another heating source for browning and crisping. Major oven manufacturers have marketed high-speed ovens using microwave/light and microwave/convective heat. One manufacturer has also developed a vending machine that heats frozen meals using microwave/jet impingement.

In software, the major difference among the ovens is in the ways of controlling the cooking process. Various cooking algorithms have been developed to control the heating sources and heating time. In the future, software development will likely incorporate more food science and technology (to develop cooking instructions, provide nutritional information, etc.), better user interface technology, and the use of fuzzy logic.



**Figure 5** An intelligent oven.

## **B.** Intelligent Ovens

In recent years, researchers have also developed the so-called intelligent ovens, which use information technology to overcome some of the limitation of the microwave oven and enhance the cooking experience of the consumer. An example of the intelligent oven is shown in Fig. 5.

The intelligent oven is a multimode oven (e.g., microwave/convective heat oven) equipped with a bar code scanner and a microprocessor (13). The oven is connected to an input/output device (such as a touch screen) and the Internet. Here are some advantages of the oven:

- 1. As mentioned earlier, the heating instructions on food packages are intentionally vague to accommodate the many types of microwave ovens on the market. The intelligent oven can overcome this problem. By scanning the bar code on the food package, its microprocessor is able to generate the precise heating instructions that match the food and the oven.
- 2. The scanning eliminates the need of entering the heating instructions manually. This is particular convenient when the instructions involves complicated multiple heating sequence, especially for multimode ovens. This feature is also helpful for visual impaired consumers.
- 3. The Internet connection allows the access of information relating to nutrition, product recall, allergenic ingredients, and so on.

# VI. SUMMARY

While microwavable frozen food and meals have become an integral part of our lifestyle, improvements are still needed to continue to justify their place in the freezer case. Although microwavable frozen products can provide the consumer with convenience, they often fail to impress the consumer with taste and texture. There are many technical and economical challenges for developing new and improved microwavable frozen products. To meet these challenges, the industry (food manufacturers, packaging suppliers, and oven manufacturers) and academia should work more closely together—to innovate and develop better microwavable food products that are more tasty, healthy, and convenient to use.

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# 33 Safety of Frozen Foods

Phil J. Bremer
University of Otago, Dunedin, New Zealand

Stephen C. Ridley

University of Wisconsin-River Falls, River Falls, Wisconsin, U.S.A.

#### I. INTRODUCTION

Foods and other biological tissues become frozen when nearly all the intra- and intercellular water is converted to ice, generally at temperatures slightly less than  $0^{\circ}$ C. From a commercial standpoint, however, frozen foods are those that are processed and maintained at temperatures below  $-18^{\circ}$ C (1). Since temperatures of  $-8^{\circ}$ C or lower are generally sufficient to prevent the growth of microorganisms (2), freezing is so effective in controlling microbial activity that chemical (oxidative and enzymatic) or physical changes (ice crystal formation and tissue dehydration) rather than microbiological activity are generally the limiting factors for frozen food shelf-life.

While microorganisms will not grow in frozen foods, this does not necessarily ensure microbiological safety. Ready-to-eat frozen foods such as ice cream and salad shrimp may carry a burden of risk similar to that of their unfrozen counterparts and therefore need to be prepared using processes and procedures conforming to appropriate regulatory guidelines and good manufacturing practices (GMPs). When assessing risk potential, from a regulatory and HACCP perspective, frozen foods are more or less equivalent to unfrozen products.

Freezing cannot be relied upon to ensure the safety of frozen foods since human pathogens surviving in the foods are likely to be infective on thawing. Furthermore, contaminated frozen foods may act as sources of contamination to food processing equipment, food plant workers, and other food products. The impact of freezing and frozen storage on the survival of pathogens and other microorganisms is dependent on many extrinsic and intrinsic factors such as temperature, storage time, freezing and thawing rates, composition of the food matrix, species of interest, and their physiological status prior to freezing.

Concerns about the occurrence of pathogens in frozen foods are increasing as consumers, processors, and regulatory authorities seek higher microbiological standards for the foods they import, process, and consume. Higher standards are paradoxically becoming harder to achieve for some food products because of increasing consumer

demand for more minimally processed foods with lower levels of salt, sugar, and other additives. As the food processing industry addresses these challenges, the importance of stringent control of plant and food hygiene and of the time and temperature regimes involved in the production of frozen food products is becoming more and more critical.

The physical effects of freezing on microorganisms have been addressed in Chapter 5 of this volume. The focus of this chapter is food safety and the concerns associated with frozen foods, including, pathogenic organisms and toxins, the impact of freezing and thawing on the recovery and detection of pathogens, and practical steps that can be taken to ensure the safety of frozen foods.

#### II. PATHOGENS ASSOCIATED WITH FROZEN FOODS

Although food-borne pathogens are frequently detected in frozen foods during inspections and routine testing, there have been surprisingly few documented outbreaks of food-borne illness associated with their consumption. The safety of frozen food products to a great extent reflects the quality and safety of the foods and ingredients prior to freezing. The inadvertent use of poor quality raw ingredients, contaminated processing equipment, inadequate pasteurization or sterilization processes, and the failure to prevent postpasteurization contamination can result in frozen products containing pathogenic organisms or their by-products at unacceptable levels.

With regard to safety, the main issues of concern are the initial levels of contamination by pathogens or by their toxins and the impact of freezing on cell survival and viability. The organisms of concern include both infectious and toxin-producing bacteria, viruses, and parasites such as nematodes tapeworms and roundworms. There have been no published reports of safety issues with molds or yeasts in frozen foods. This section discusses the pathogens that have been involved in outbreaks and those that may cause food-borne illness through the consumption of frozen foods.

## A. Bacteria

In San Jose, Costa Rica, retail samples of homemade (35 samples) and commercially produced (30 samples) ice cream were purchased and tested for the presence of pathogens. It was determined that 18 (51.4%) of the homemade and 8 (26.7%) of the retail samples contained Escherichia coli. Eight samples of homemade ice cream contained Listeria, and half the 16 Listeria isolates recovered were Listeria monocytogenes; the remainder were L. innocua. Salmonella was not detected (3). In a similar trial, commercial ice creams (30 samples) from Mumbai, India, were examined. Staphylococcus aureus was found in all samples, while Bacillus cereus and Yersina enterocolitica were found in 10 and 11 of the samples, respectively. Although Listeria was detected in 23 of the samples, L. monocytogenes was found in only 1 sample, and Salmonella was not detected (4). Listeria monocytogenes has been isolated from several brands of ice cream (5–9), and consumption of contaminated ice cream has been implicated in a case of human listeriosis (10). Obviously, the use of poor quality ingredients and inadequate pasteurization or sanitation can result in ice cream becoming contaminated. In Karachi, Pakistan, the consumption of ice cream was identified (along with eating at roadside food stands and drinking water at a work site) as a major risk factor associated with developing typhoid fever (11).

One of the most highly publicized incidents of food-borne disease occurred in 1994 in the United States when the Minnesota Department of Health detected a precipitate rise in the number of reports of Salmonella Enteritidis infections. When a case-control study implicated nationally distributed Schwan's ice cream in the outbreak, a national recall and a customer surveillance plan were immediately put in place. Sampling of the product and processing environs implicated tanker trailers that had previously carried unpasteurized eggs immediately prior to transportation of pasteurized ice cream mix as the likely source of contamination. From the data obtained, it was estimated that S. Enteritidis gastroenteritis developed in 224,000 (6.6%) of the individuals who had consumed the contaminated ice cream (12). Quantitative analysis of contaminated samples indicated that the number of S. Enteritidis cells per serving of ice cream (65 g) was 25. Based on consumption of a single sundae cone (73 g, prepackaged), which caused severe illness in an eight-year-old boy and moderate to mild illness in the adult parents, the infective dose was estimated to be no more than 28 cells (13).

A study of retail meats, poultry, and fish obtained over a 2 year period in Dublin, Ireland, revealed that 97% of raw frozen meats contained *Listeria*. The most common species encountered were *L. innocua* (38%) and *L. monocytogenes* (11%), and there was a much higher occurrence of *Listeria* in frozen (97%) than in fresh meats (45% to 85%) (14). Other studies have reported high percentages of *Listeria* in frozen meats (15, 16) and in seafood (17); Wang et al. (16) also reported a higher incidence for *Listeria* in frozen (88.6%) than in fresh (22.8%) meats. The incidence of *Campylobacter jejuni* in chilled and frozen chicken carcasses obtained from 21 retail stores over a 3 month period was reported to be 70% (22 tested) and 20% (37 tested), respectively (18).

The occurrence of low levels of pathogens in frozen meats and fin fish products has traditionally caused little concern because of the expectation that such products would receive a bactericidal heat treatment (cooking) prior to being consumed. While this is generally still the case, the presence of pathogens in previously frozen products that subsequently receive inadequate heating can result in disease. Therefore restaurants, food retailers, and food processing companies are increasingly striving for more stringent microbial standards in the products that they purchase.

#### **B.** Bacterial Toxins

Bacteria such as Clostridium botulinum and Staphylococcus aureus can produce extracellular toxins during growth in foods. Frozen storage has been reported to have no effect on the titer of preformed type E toxin from C. botulinum added to canned salmon and corn stored at  $-15^{\circ}$ C for up to 264 days (19) or type A toxin added to tomato or mushroom soup, beef pie filling, or phosphate buffer stored at  $-20^{\circ}$ C for up to 180 days (20). The heat-stable staphylococcal enterotoxins are also unaffected by freezing, and illness can result if frozen foods containing preformed toxins are ingested (21, 2). Bacterial species including Morganella morganii can convert the amino acid histidine, which naturally occurs in some species of fish and other foods, to histamine, which once formed is not affected by frozen storage. Ingestion of preformed histamine can result in an allergiclike response known as histamine or scombroid poisoning (22).

For microbial toxins to be present in sufficient concentrations to cause illness, a frozen food or a frozen food ingredient would have been subjected to temperature abuse either by improper cooling or by storage at elevated temperatures prior to freezing. While the potential exists for preformed toxins to cause illness, there is little evidence to suggest their involvement in large numbers of food poisoning incidents associated with the consumption of frozen foods.

#### C. Viruses

There is ample evidence that viruses in contaminated seafood readily survive processing by freezing. DiGirolamo et al. (23) demonstrated that polio virus inoculated into Pacific and Olympia oysters slowly lost viability during frozen storage, but 10% of the population remained viabile after storage for 12 weeks at  $-17.5^{\circ}$ C. In a similar experiment, Greening et al. (24) allowed New Zealand Green Lipped mussels to accumulate poliovirus through filter feeding in a closed system. Some of the mussels were subsequently frozen, put in frozen storage at  $-20^{\circ}$ C, and tested for viability by plaque assay and by reverse transcription polymerase chain reaction followed by dot blot hybridization. The percentage of infective virus units after storage for 7, 14, and 28 days was 66%, 53%, and 44%, respectively.

An incident of viral infection following the consumption of a frozen product occurred in Philadelphia in 1987 when over 200 university students and football team members exhibited symptoms typical of Norwalk viral gastroenteritis. Ice used in soft drinks was identified as the most likely source of the virus (25). In an outbreak of gastroenteritis in employees of a large company in Helsinki, Calicivirus was identified as the most likely cause, with the source of the virus being imported frozen raspberries used to prepare a dressing in the company kitchen. (26).

Other reports of food poisoning episodes due to viral contamination of frozen foods are limited, but it is assumed that with the increase in awareness of the role of viruses in food-borne disease, an increased incidence of discovery will result from improved methodologies and investigations in which specific viral agents are targeted. It is more or less clear that frozen foods must be considered as a potential reservoir for several kinds of infective viruses, including hepatitis A and E, Norwalk, rotovirus, and polio. This is an area of food microbiology that requires considerably more research effort in the future.

#### D. Parasites

In contrast to most bacteria, fungi, and viruses, which are generally resistant to the effects of freezing, frozen storage, and thawing, there are several human parasites including certain protozoans, helminthes, nematodes, and coccidia that show poor tolerance, and in some cases they can be totally eliminated from foods by properly applied freezing protocols. Most notable in this regard is *Trichinella sprialis*, the parasitic worm (nematode) that causes the human illness trichinosis, through the consumption of undercooked, infected pork or wild game. Although trichinosis is thought to be rather uncommon in most industrialized countries, a study by Zimmerman et al. (27) reported that about 2.2% of the U.S. population was infected.

In 1960 the U.S. Department of Agriculture (USDA) began a certification program to ensure that raw or minimally cooked pork to be used in ready-to-eat products would be free of *Trichinella* larvae. One method involves freezing pork to a center temperature of  $-30^{\circ}$ C and holding for a minimum of 16 hours. Other approved methods involve higher freezing temperatures (from  $-29^{\circ}$ C to  $-15^{\circ}$ C) with storage for increasing periods of time. The approved temperature–time combinations vary according to the thickness of pork cuts. All approved methods guarantee complete lethality (28). The USDA has also provided similar recommendations for home processed pork. These recommendations do not appear to be effective for wild game meat infected with the Arctic strain or subspecies of *T. spiralis* (29).

Freezing is also effective in the elimination of *Anisakis simplex* larvae from fish. *Anisakis simplex* is a marine nematode (roundworm) that was first reported to have infected humans in the Netherlands in 1955 (30). In 1968 in the Netherlands raw herring was required to be frozen at  $-20^{\circ}$ C for at least 24 hours before distribution (31). There is little information on the effects of freezing on other fish-borne parasites such as tapeworms (Cestodes) and flukes (Trematodes).

Human cysticercosis results from eating meat containing viable larvae of one of several species of the tapeworm *Taenia*. *T. saginata*, *T. hydatigena*, *T. ovis*, and *T. solium* are traditionally associated with beef, sheep, goat, and swine carcasses (32). It has been reported that cysticercosis of bovine origin can be prevented by keeping meat from infected animals frozen for 48 hours prior to consumption (33). Kim (34) reported, that the larvae of *T. saginata* (beef tapeworm) are inactivated by freezing and holding at  $-10^{\circ}$ C for 10 days or at  $-18^{\circ}$ C for 5 days. Likewise, *T. solium* larvae (pork tapeworm) can be inactivated by freezing meat and holding it at  $-10^{\circ}$ C for at least 14 days.

The effects of freezing foods on protozoan viability are still unclear. *Toxoplasma gondii* is a protozoan that has historically infected a large proportion of people in the U.S. It has been reported that freezing meat infected with T. *gondii* has an inconsistent effect on the human infectivity of Toxoplasma. Differences in viability were considered to be due to the cold-hardiness of different strains (35), and the authors recommended that freezing should not be considered as a means of control. Other authors have stated that freezing of meat to  $-13^{\circ}$ C will usually render T. *gondii* cysts nonviable (36, 37). Oocysts of *Cryptosporidium parvum*, a parasitic protozoan, have been reported to be susceptible to freezing. There is currently no information on the susceptibility of *Cyclospora* spp. (38).

Although approved freezing conditions have proved to be effective in eliminating viable forms of some parasites from meat and fish, it has been pointed out that freezing may be less cost-effective than other equally effective methods, including microscopic inspection of pooled digested samples (29).

#### III. EFFECTS OF FREEZING ON MICROORGANISMS

Microorganisms differ in their sensitivity to freezing and frozen storage, the effects of which fall on a continuum from no injury through to sublethal injury to death (39, 40). There have been no reports on the effects of freezing on bacteria in the viable but not culturable state. Typical responses of various types of microorganisms are shown in Table 1.

Because of inequities in the ability of various types of microorganisms to tolerate freezing, the process itself exerts a selective effect on the microflora of frozen foods. For example, the microflora of a sample of raw meat before freezing was estimated to number  $3.85 \times 10^5$  cell g<sup>-1</sup> and consisted of 15% gram-positive and 85% gram-negative bacteria. After freezing, the number of surviving cells was reduced only slightly to  $7.7 \times 10^4$  cell g<sup>-1</sup>, but the population was composed of 70% gram-positive and only 30% gram-negative bacteria (41).

In general, gram-positive bacteria are more resistant to freezing than gram-negative bacteria, and cocci are more tolerant than bacilli. Yeasts and molds are typically more resistant to freezing than bacteria, perhaps because of their enhanced ability to survive under conditions of low water activity (42, 43).

**Table 1** Effects of Freezing on Microorganisms and Their By-Products

Response	Example
Survival under virtually all conditions of freezing and thawing	Bacterial and fungal spores, vegetative cells of gram-positive cocci such as micrococci, streptococci, and staphylococci enterotoxins, histamine, many viruses
Resistance to freezing, but may be metabolically injured during frozen storage and/or thawing	Gram-positive rods such as <i>Listeria</i> monocytogenes, <i>Bacillus</i> , <i>Clostridium</i> , and <i>Lactobacillus</i> species, yeasts
Sensitive to freezing, frozen storage, and thawing under some, but not all conditions. If present in sufficiently high numbers before freezing may survive long periods of frozen storage	Gram-negative organisms such as Escherichia, Salmonella, Serratia, Pseudomonas, and Vibrio. The gram-positive Clostridium perfrigens
Inactivated under nearly all freezing and thawing conditions	Many parasites such as <i>Trichinella</i> , <i>Taenia</i> , <i>Anisakis</i> , and <i>Toxoplasma</i>

## A. Damage to Microorganisms Due to the Freezing Process

In practice it is difficult to distinguish between the effects of freezing, frozen storage, and thawing, since the parameters used to define viability and injury are usually linked to the ability of an organism to reproduce or exhibit respiratory activity. Virtually all of the research on the effects of freezing on microorganism survival has been conducted on organisms exposed to one or more freeze/thaw cycles. This topic has been covered in detail in Chapter 5 of this handbook and will therefore be reviewed only briefly here.

The lethal effects of extreme cold on microorganisms may be direct and more or less immediate during the processes of freezing and thawing. Lethality may also be delayed and result from prolonged storage at subfreezing temperatures (40).

The mechanisms of damage to microbial cells by the freezing process was summarized by Marth (40) as being due to four distinct processes (Table 2).

The injury or death of a microorganism upon sudden chilling is referred to as cold shock. Cold shock was first referenced by Sherman and Albus in 1923 (44) for *E. coli* and is now known to occur in a variety of both gram-negative and gram-positive cells (45). Cold shock is accompanied by the release of a number of low molecular weight intracellular solutes including nucleotides, amino acids, ultraviolet absorbing materials,

 Table 2
 Process and Site of Damage Due to Freezing

Process	Damage
Rapid temperature reduction (cold shock) Extracellular ice crystal formation Intracellular ice crystal formation Concentration of solutes	Increased permeability of cell membranes Dehydration—minor damage Mechanical damage Osmotic effects may increase reaction rates, cause changes in pH, and result in changes in the concentrations of various ionic species that may have toxic effects

and ATP (45). Gram-negative bacteria, particularly those in an exponential growth phase, are the most susceptible to the deleterious effects of sudden temperature reductions. There is evidence that the response to cold shock is mediated by growth temperature, since cells grown at temperatures nearer the lower extremes of the growth range are more resistant. Physiologically, cold shock resistance has been associated with an increase in the concentration of unsaturated fatty acids in the membrane lipid fraction. The damaging effects of cold shock are also diminished by higher concentrations of cells in the freezing menstrum. (46).

If there is significant extracellular ice crystal formation during the freezing process, water can migrate from the bacterial cell to the medium, which results in cell dehydration and the concentration of intracellular solutes.

The immediate death of cells during freezing while being predominantly associated with cold shock may also be caused by the development of intracellular ice crystals. Relatively large cells, such as yeasts and fungal spores, seem to be more seriously affected by ice crystals than are bacteria (40). Intracellular crystals are believed to distort the cell membrane resulting in loss of integrity, which leads to the leakage of intracellular constituents and the failure of the cell to maintain its internal environment. However, with normal rapid freezing methods that are typical of commercial frozen food production, ice crystal formation is greatly reduced and is probably not a major factor in microbial cell death or damage.

As well as causing direct damage, freezing changes the composition of the medium surrounding the cell, which can affect cell survival. During freezing, bacterial cells behave like solute molecules and can become partitioned and concentrated in the unfrozen part of the solution as ice crystals form. Freeze concentration can affect reaction rates, pH, and the ionic strength of the unfrozen liquid, resulting in cell death during storage. For example, this process can alter the pH, in some foods by as much as 2 pH units. Further, as the microorganisms are exposed to high solute concentrations, water is transferred from the cell to the medium, resulting in cell dehydration. Increases in the ionic strength of the unfrozen phase can cause denaturation of macromolecules such as DNA and proteins. Freezing can also result in the loss of cytoplasmic gases such as oxygen and carbon dioxide, and the suppression of the respiratory activity of aerobic cells. However, since the decrease in temperature has a dramatic impact on the cells' metabolic activity, it is difficult to determine the comparative effect of these processes (40, 42, 43, 45).

#### B. The Effect of Freezing Conditions on Microorganisms

Many intrinsic and extrinsic factors inherent in the freezing process affect the food microflora. In general, the survival of microorganisms in frozen foods is enhanced by lower storage temperatures, with storage at temperatures near the freezing point resulting in the highest degree of death or injury (40). Cell survival is improved when the storage temperature is stable during storage.

Freezing rate is also an important determinant of microbial viability. During slow freezing, ice crystals form in the medium outside the cells. As the cytoplasm in cells becomes supercooled at -5 to  $-10^{\circ}$ C, the vapor pressure in the cells exceeds that of the ice crystals, resulting in a loss of water from cells. Additionally, as the extracellular water freezes, the concentration of solutes increases, resulting in further loss of water from cells owing to plasmolysis. Such slow freezing exposes cells to prolonged osmotic effects, thus increasing the chance of injury (47). With faster freezing rates, the time of exposure to damaging osmotic effects is reduced, thereby improving the chances of survival.

When freezing rates are relatively fast, intracellular ice crystals form that may injure the cell and reduce chances of survival. For example, at rates of freezing within the range of 1°C to 10°C per minute, cell viability is enhanced, owing to shorter contact time of the organisms to the highly concentrated solutes present in the unfrozen water. In contrast viability has been reported to decrease at rates in the range of 10°C to 100°C per minute owing to the formation of intracellular ice crystals that destroy the cell membranes. At freezing rates ranging from 100°C to 1000°C per minute (encountered in cryogenic freezing) viability is enhanced owing to a reduction in ice crystal formation (48).

The composition of the food or the medium also influences the effect of freezing on microbial cells. The presence of sodium chloride reduces the freezing point and thereby extends the time during which cells are exposed to high concentrations of solutes prior to the beginning of freezing. Other compounds, such as glycerol, sucrose, gelatin, and proteins, generally have a protective effect (47). Chemical compounds that generally inhibit bacterial growth have been reported to show enhanced effectiveness when combined with freezing. Cultures of Salmonella Typhimurium and other gram-negative bacteria were exposed to various forms of laurate (sodium laurate, sodium lauryl sulfate, monolaurin, polyoxyethylene sorbitan monolaurate, sorbitan monolaurate) at low concentrations during freezing in nutrient broth. The concentrations selected for the experiment had previously shown no effect on the growth of target cultures that had not been subjected to freezing. Of the chemicals tested, sodium laurate was the most lethal. Laurates were considered to cause direct damage to cells during freezing, since they showed no effect in reducing viability when added after thawing. The lethal effects of laurates were related to freezing rate, with effectiveness being increased when the freezing rate was slow. The effect of laurate was not observed when 10 mM calcium or magnesium were supplemented into the menstruum. These cations were previously reported to form an insoluble complex with laurate such that it was unable to interact with cells. Alternatively, these cations may act to protect the cell wall or the cell membrane (49).

Smith (50) conducted chilling and freezing survival studies on several laboratory and wild strains of E. coli and Salmonella cultures in noninhibitory nutrient broth. The studies showed that cells in late lag phase or in exponential phase were more susceptible to freezing death than cells in stationary phase and that magnesium ions in the medium conferred a degree of protection from the effects of freezing. With wild isolates, considerable strain variation in the ability to survive freezing was apparent. Resistance to freezing death tended to be lost after subculturing in the laboratory. It was concluded that predictive modeling of freezing lethality in actual food systems would be difficult, since some of the factors that affect cell viability cannot be conveniently measured or accounted for outside the laboratory.

A summary of factors recognized as affecting the response of cells to freezing is presented in Table 3.

## C. Experimental Studies on the Survival of Pathogens in Frozen Foods

Over the course of the past 40 years a number of studies have determined the effects of freezing, frozen storage and thawing on the viability of food-borne pathogens. Studies have been conducted with cells in food materials, in buffers, or in microbiological media such as tryptic soy broth. The phase of growth, small changes in the composition of testing medium, and subcultures made after primary isolation all influenced cell survival. While organisms such as salmonellae, *L. monocytogenes*, and *E. coli* have been the subject of numerous studies, data available on a number of other pathogens are limited. In addition

**Table 3** Parameters That Impact on the Effect of Freezing on Microorganisms

Parameter impacting on the effect of freezing

Type and strain of organism

Population density

Nutritional status

Growth phase (lag, exponential or stationary)

Rate of growth

Composition of the freezing menstrum

Cooling rate to the freezing point of the suspension

Cooling rate from the freezing point of the suspension

Time held at low temperature

Holding temperature

Rate of warming to freezing point

Diluting environment prior to viability determination

Method of viability determination

Medium used to assess viability

Source: Ref. 45.

it is likely that the effects of freezing on bacterial cell death have been overestimated. As discussed in Sec. IV, freezing can damage cells so that they are not recoverable by traditional microbiological techniques. As more becomes known about the mechanisms of freezing damage, however, media better suited for recovery are continually being developed. Given these limitations and the multitude of intrinsic and extrinsic factors that are known to affect the freezing process (Table 3), caution must be exercised in extrapolating the results of pure culture testing to the prediction of the behavior of similar organisms in frozen foods.

In general, however, it can be concluded that although freezing causes a reduction in bacterial cell viability and possibly a loss of infectivity of some viruses, in most cases freezing technology cannot be used as a substitute for lethal technologies such as thermal processing or ionizing radiation for ensuring the safety of processed foods. The following sections summarize some of the freezing studies performed on various pathogens.

#### 1. Escherichia coli

Escherichia coli has been reported to survive well in chilled and frozen foods. In frozen foods there is usually an initial decrease followed by a much slower death rate during storage. Semancheck and Golden (51) reported that holding E. coli O157:H7 for 7 months at -20°C in peptone water resulted in a 4 log to 6 log CFU mL<sup>-1</sup> reduction in viability. Escherichia coli O157:H7 numbers in ground beef patties during 9 months storage at -20°C decreased by only 0.5 log CFU g<sup>-1</sup> (52) and by less than 50% in ground chicken breast meat stored at -20°C over 18 months (53). Ansay et al. (54) studied the survival of E. coli O157:H7 during low-temperature storage in ground beef patties and found that tempering (preincubation of inoculated patties at 15°C for 4 hours) prior to storage at -2°C was detrimental to survival. The numbers of E. coli O157:H7 in untempered and tempered patties held at -2°C for 4 weeks decreased by 1.5 log CFU g<sup>-1</sup> and 2.9 log CFU g<sup>-1</sup>, respectively. In ground beef patties stored at -20°C, cell numbers decreased by approximately 2 log CFU g<sup>-1</sup> after 1 year (54).

Factors influencing the survival of E. coli O157:H7 during freezing and thawing in ground meat patties held at  $-20^{\circ}$ C for 24 hours were reported to include strain (4 strains tested), the thawing method (held at either  $4^{\circ}$ C or  $23^{\circ}$ C or microwaved), and the method used to enumerate survivors (MPN or plating onto two different agars). Overall the death of E. coli in the frozen and thawed patties ranged from 0.62 log to 2.52 log, and the authors concluded that even though more than 99% of cells may die in some situations, freezing and thawing cannot be regarded as a significant intervention strategy for control of E. coli O157:H7 in ground beef (55).

Jackson et al. (56) reported that  $E.\ coli$  O157:H7 demonstrated increased resistance to heat induced lethality after being frozen at  $-18^{\circ}\mathrm{C}$  for 8 days in ground beef patties. When frozen patties were subsequently held at  $21^{\circ}\mathrm{C}$  or  $30^{\circ}\mathrm{C}$  to simulate temperature abuse, the organism appeared to lose the heat resistance gained during freezing. These observations led to the conclusion that ground beef storage and holding temperatures encountered in food service operations can significantly influence the ability of pathogenic  $E.\ coli$  to survive heating (56). In a similar study involving apple juice, the numbers of  $E.\ coli$  cells in neutralized apple juice did not change during 21 days of frozen storage at  $-20^{\circ}\mathrm{C}$ ; numbers in acidified samples (pH 4.2) decreased by only 1 to 3 logs depending on the strain tested (57).

Grzadkowska and Griffiths (58) conducted a detailed study to compare the survival of strains of  $E.\ coli$  O157:H7 with nonpathogenic strains of  $E.\ coli$  in a variety of food materials and in laboratory media following a lowering of the temperature from that of optimum growth to  $20^{\circ}\text{C}$  (cold shock) prior to subsequent freezing at  $-18^{\circ}\text{C}$ . Survival under conditions of cold shock followed by freezing was referred to as cryotolerance. The fact that cold-shocked  $E.\ coli$  O157:H7 showed a 25% to 30% increase in their ability to survive frozen storage for 24 hours at  $-18^{\circ}\text{C}$  when compared with non-cold-shocked cells demonstrated the efficacy of cold temperatures as a survival strategy. In contrast, non-O157:H7 strains showed cryotolerance of only 5%.

In the experiment described, the suspending medium affected the cold-shock response in all the bacterial strains studied. The largest cold-shock effect occurred in Brain Heart Infusion Broth, and it was also evident in frozen apple juice (pH 3.53). Cryotolerance was not observed, however, when cells were suspended in frozen yogurt or ground beef (58).

In addition, cryotolerance in strains of E. coli O157:H7 was not evident, in an experiment described by Semancheck and Golden (51), who showed that strains held at  $10^{\circ}$ C were actually more sensitive to freeze inactivation than strains cultured at  $37^{\circ}$ C prior to freezing in peptone water.

In cases where cryotolerance has been observed, it has been assumed from the work of Goldstein et al. (59), that a cold-shock temperature of 20°C resulted in the cellular production of cold-shock proteins (CSPs), which conferred resistance to the some of the lethal effects of freezing.

#### 2. Listeria monocytogenes

Most studies have indicated that all strains of *Listeria monocytogenes* are quite resistant to the damaging effects of freezing. Golden et al. (60) inoculated early stationary phase cells of 4 strains into tryptose phosphate broth to investigate inactivation and injury. The viability of all 4 test strains was not appreciably reduced after 14 days at  $-18^{\circ}$ C, based on recovery using nonselective media. Recovery was reduced, however, after adding

increasing concentrations of sodium chloride ranging from 2% to 8%, indicating that sublethal injury had occurred.

In ground beef, ground turkey, frankfurters, canned corn, and ice cream mix, which all had a pH > 5.8, L. monocytogenes survived freezing, was not injured, and was quantitatively recovered using standard Listeria recovery media after 14 weeks at  $-18^{\circ}$ C. In tomato soup (pH = 4.74), held under the same conditions, Listeria demonstrated sublethal injury and could not be quantitatively recovered using selective media. The authors concluded that the survival of L. monocytogenes during frozen storage was related to the pH of the food and that for most foods, freezing of samples prior to analysis for L. monocytogenes should not hamper quantitative determination of the organism (61).

Cells of L. monocytogenes have been shown to be more resistant to death and injury during freezing and storage at  $-18^{\circ}$ C when they are suspended in milk or tryptose broth rather than in phosphate buffer solution (62). Lactose (2%) and milk fat (2%) resulted in protection against cell death and metabolic injury, lactose giving protection against injury for 5 months as compared to 4 weeks with milk fat (63, 64). It was concluded that frozen dairy foods that contain high concentrations of compounds such as lactose, casein, and milk fat are likely to protect L. monocytogenes during frozen storage and that care should be taken to prevent contamination of such products with Listeria during processing (63).

#### 3. Salmonellae

Numerous reports of the isolation of salmonellae from frozen foods such as ice cream provide ample evidence of the ability of salmonellae to survive in frozen products. In minced chicken breast (pH 5.8), 60% to 83% of *Salmonella* cells survived storage at  $-20^{\circ}$ C for 126 days, while at temperatures of either  $-2^{\circ}$ C or  $-5^{\circ}$ C only 1.3% and 5.8% survived after 5 days (65). The effects were investigated (66) of 10% (w/v) salt, trisodium phosphate (TSP), sodium tripolyphosphate (STPP), and tetrapotasium pyrophosphate (TKPP) washes on the survival of *Salmonella* Typhimurium associated with chicken breast patties during frozen storage ( $-20^{\circ}$ C) or after 3 freeze—thaw cycles. After 3 freeze—thaw cycles and 10 months of storage at  $-20^{\circ}$ C, greater reductions were noted for all samples washed with salt, TSP, STPP, and TKPP compared to the control. Reductions in cell numbers were greatest in TSP treated cells. Reductions were log 4.92 CFU patty<sup>-1</sup> after 3 freeze—thaw cycles and log 7.15 CFU patty<sup>-1</sup> after 10 months at  $-20^{\circ}$ C. In all cases, *S.* Typhimurium could still be recovered from the patties (66).

White and Hall (67) studied the effects of storage temperature abuse on the survival of salmonellae in chicken and beef substrates by means of thawing and refreezing experiments. They reported that viable counts of *Salmonella* Typhimurium declined during the first 4 hours of thawing at 20°C. Another serotype, *S.* Hadar, responded differently, demonstrating an active growth response during the earliest stages of thawing. Reductions in the populations due to refreezing were influenced by the duration of the thawing period. Refreezing the substrates to  $-18^{\circ}$ C following thawing periods of 4, 8, and 24 hours at 27°C resulted in population reductions of 84% to 36%, *S.* Typhimurium being more severely affected. The results of these experiments and those from similar work by Olson et al. (68) illustrate the inherent difficulties in predicting or modeling growth responses resulting from complex circumstances involving mixed microbial populations during freezing and thawing cycles varying in terms of temperature and time. These results also have implications for laboratory handling of frozen foods that need to be thawed prior to testing.

## 4. Campylobacter

The impact of freezing on the survival of *Campylobacter* spp. appears to be quite variable. Freezing has been reported to cause an initial 1 log reduction in numbers of *C. jejunui* followed by a gradual reduction during storage. On chicken livers inoculated with log 5.00 *C. jejuni*  $g^{-1}$  and held at either  $-20^{\circ}$ C or  $-70^{\circ}$ C, numbers initially dropped to between log 3.3 to log  $2.3 g^{-1}$  and log 4.74 to  $3.65 g^{-1}$ , respectively. After 84 days, the numbers of cells detected at  $-20^{\circ}$ C and  $-70^{\circ}$ C were log 1.74 and log  $4.4 g^{-1}$ , respectively (69). A 3 log  $g^{-1}$  reduction in *C. jejuni* numbers was reported for contaminated minced beef held at  $-15^{\circ}$ C for 14 days (70) and a 1 log  $g^{-1}$  reduction was reported for hamburgers held at  $-18^{\circ}$ C for 7 days (18).

## 5. Staphyloccus aureus

Freezing, especially slow freezing, has been reported to reduce slightly the numbers of viable *Staphyloccus aureus* and to result in metabolic injury, thereby reducing the ability to grow in selective media (71). However, like most gram-positive bacteria, *S. aureus* is only slightly affected by frozen storage, with reductions in cell numbers on beef or chicken held at  $-18^{\circ}$ C for 6 months being only in the range of 0.1 log g<sup>-1</sup> to 1.1 log g<sup>-1</sup> (66). These authors also showed that viable counts of *Staphylococcus aureus* were not reduced by refreezing after 4 and 8 hours of thawing except when the population of the competing microflora in chicken originally outnumbered *S. aureus* by a factor of 1000. In this case, *S. aureus* counts decreased by 1 log to 2 log g<sup>-1</sup> after refreezing.

## 6. Vibrio parahaemolyticus

The survival time for *Vibrio parahaemolyticus* in frozen homogenates of oyster meat has been reported to be dependent on both the temperature of the refrigerated or frozen storage and the initial levels of bacteria present (72). At  $0^{\circ}$ C,  $-18^{\circ}$ C, and  $-24^{\circ}$ C, the calculated time required to reduce an initial inoculum of  $10^{5}$  CFU per  $g^{-1}$  to less than 1 per gram was 178, 105, and 134 days, respectively. The authors suggested that prolonged frozen storage may be considered as a means of reducing the *V. parahaemolyticus* hazards in seafood (72). Previously this organism was reported to have experienced very little decline in numbers (slightly more than 2 logs from an initial population of  $10^{6}$  CFU  $g^{-1}$ ) during storage of contaminated oysters for 82 days at  $-30^{\circ}$ C (73). Indeed, there was little further decline up to 130 days, when the experiment was terminated. During storage at  $-15^{\circ}$ C, there was a 3 log reduction during the same period. Survival of *V. parahaemolyticus* in fish fillets and in crabmeat was less than in oysters (73).

#### 7. Yersinia enterocolitica

Yersinia enterocolitica is a psychrotrophic organism that has been reported to grow at temperatures as low as  $-5^{\circ}$ C (74). Strains isolated from polar marine environments survived for up to 54 days at  $-1.8^{\circ}$ C at a salinity of 34.5 parts per thousand, though the percentage of injured cells increased with time (75).

#### 8. Shigellae

Shigellae have been reported to be recoverable from neutral foods such as butter and margarine after more than 100 days at  $-20^{\circ}$ C (76).

#### 9. Aeromonas

Abeyta et al. (77) examined samples of oysters that had been frozen at  $-72^{\circ}$ C for  $1\frac{1}{2}$  years for the presence of viable pathogenic *Aeromonas hydrophila*. The fresh oysters had previously been implicated in an outbreak of diarrheal illness of unknown cause. Low levels of various strains of pathogenic *A. hydrophilia* were recovered using a variety of commonly employed laboratory media.

#### 10. Spores

Bacterial and fungal spores are extremely resistant to freezing and storage at subzero temperatures, with viability exceeding 90% (43). However, if foods are consumed frozen or if they are thawed and held at sufficiently low or high temperatures to prevent spore germination, their presence in the food is not of particular concern. Fairhead et al. (78) showed that small acid-soluble proteins bound to DNA protected spores of *Bacillus subtilis* from being killed by freeze drying. Dormant spores of a wild-type *B. subtilis* were resistant to eight cycles of freeze drying; compare these similar spores that lacked two DNA binding proteins (small acid-soluble  $\alpha$  and  $\beta$ ) and experienced a 90% reduction in viability after 3 or 4 cycles. In the latter case, significant DNA mutation was reported to have accompanied spore death. The authors postulated a role for  $\alpha/\beta$  small acid-soluble proteins in spore resistance and survival of freeze-drying in the environment.

#### IV. SUBLETHAL INJURY

Various treatments related to food processing such as heating, drying, freezing, irradiation, changes in pH, or addition of preservatives may induce changes in bacterial physiology so that cells become injured to the extent that they are unable to grow under as wide a range of physical and chemical conditions as uninjured cells. Such cells are said to be sublethally injured (79, 80).

Injury due to freezing has been reported to be caused by a sudden drop in temperature, ice formation, and/or increased solute concentration and results in a loss of viability, leakage of cellular materials, increased nutritional requirements, sensitivity to surfactants, reduced resistance to environmental stress, extended lag phase, and decreased resistance to radiation. For example, when a culture of *E. coli* O157:H7 was frozen and held at  $-5^{\circ}$ C,  $-18^{\circ}$ C, or  $-28^{\circ}$ C and subsequently thawed, cells demonstrated increased susceptibility to crystal violet, bile salt, sodium chloride, and ethanol. The culture frozen and stored at  $-18^{\circ}$ C was more susceptible than cells frozen and stored at  $-5^{\circ}$ C or  $-28^{\circ}$ C, and susceptibility increased with storage time regardless of temperature (81). When *L. monocytogenes* cells were frozen and held at a temperature between  $-9^{\circ}$ C and  $-11^{\circ}$ C, the number of injured cells after 24 hours ranged from 44% to 64%, and the percentage of injured cells increased only slightly after 14 days' frozen storage (82).

Many of the culture media that are traditionally used to detect pathogens contain compounds designed to inhibit competing microorganisms and to select for the organism of interest. These compounds can significantly reduce the ability of media to support the repair and growth of injured cells. Unless a medium is optimized to support both injured and uninjured cells, or unless, as is often the case, a preenrichment step incorporating a nonselective medium is used, injured cells will not be recovered, and the incidence of a pathogen will be underreported.

Jay (83) stated that while the recognition of sublethal stresses on food-borne microorganisms and their effect on growth under varying conditions dates back to the turn of the 20th century, a full practical appreciation of this phenomenon was not forthcoming until the 1960s. Gunderson and Rose (84) noted that the numbers of coliforms from frozen chicken products that grew on Violet Red Bile Agar (VRBA) progressively decreased with increasing storage time. In later experiments, where salmonellae were inoculated onto food that was subsequently frozen, it was found that more organisms could be recovered on highly nutritive nonselective media than on selective media (85). The importance of the isolation medium in recovering stressed cells was later noted by others (86, 87).

Given suitable temperatures and nutrients, most freeze-injured cells will regain their original characteristics within several hours. Many authors, for example, have reported that *L. monocytogenes* becomes sublethally injured upon exposure to freezing stress and that this injury is reversible (82, 60, 64, 61). The consequences of freeze injury are not believed to be transmitted during cell division, indicating that freezing does not cause permanent changes to the cell's genetic material.

One of the commonly observed effects of sublethal injury is leakage of low-molecular-weight cellular material, including peptides and amino acids, indicating damage to the cell membrane. Where large numbers of cells are present, this leakage is thought to provide some degree of cryoprotection. Sensitivity to membrane-active inhibitory agents, such as the surface-active compounds used as selective agents in microbiological media, is increased. This has been taken to indicate that freezing causes major conformational and functional changes in the cellular structures controlling membrane permeability.

It has been noted that the temperature regime to which cells are exposed prior to freezing can alter an organism's ability to survive the freezing process. Because of the importance of freezing in food preservation, there has been much interest in the cold adaptation of microorganisms (88) and the response of bacteria to an abrupt decrease in the growth temperature (cold shock). After a sudden decrease in temperature, many bacteria synthesize increased amounts of small (7 kDa) proteins, the so-called cold-shock proteins (CSPs). These proteins share a high degree of similarity in a variety of grampositive and gram-negative bacteria, including *E. coli, Bacillus subtilis, Bacillus cereus, Salmonella* Enteritidis, *Salmonella* Typhimurium, and *Lactobacillus plantarum* [see Abee and Wouters (89), for a review]. CSPs are suggested to function as RNA chaperones facilitating the initiation of translation under low temperatures. While the significance of the cold-shock response is still uncertain, clear differences in microbial survival upon freezing has been reported for cells previously exposed to different cold-shock treatments (59, 90), with cold shock being postulated to enhance bacterial survival in frozen foods (89).

Mackey and Derrick (91) reported that freeze-injured  $E.\ coli$  recovered better in a nutritionally rich medium than in minimal media. This contrasted markedly with  $E.\ coli$  that had been cold shocked, which recovered better in minimal media. It was suspected that metabolically produced peroxides accumulated in rich media, but not in minimal media. In support of this hypothesis was the observation that Salmonella Typhimurium, which had been grown previously in a medium containing 30 micromoles of hydrogen peroxide per liter, was not affected by a rich medium after cold shock. It was assumed that some bacteria contain inducible systems that when activated can protect them from oxidative stress. A measurable degree of resistance to freezing injury was conferred on exponential phase cells of Salmonella Typhimurium when incubated with large numbers ( $\geq 10^8$ ) of exponential-phase gram-negative competitor cells ( $E.\ coli$ ,  $Pseudomonas\ fluorescens$  and  $Citrobacter\ freundii$ ). The degree of resistance conferred was equivalent to

that of salmonellae that were in stationary phase (92), and the phenomenon was termed stationary-phase induction. This study led to the identification of RNA polymerase sigma factor as a probable inducer of resistance to freezing damage. Sigma factor was previously known as a regulator of a group of genetic systems that contribute to virulence and to resistance to heat, osmotic stress, and oxidative damage, thus contributing to cell survival.

Repair of freeze-damaged cells occurs during the lag period, where there is intense metabolic activity involving RNA and ATP synthesis and reorganization of membrane components such as lipopolysaccharides, without accompanying growth. The *de novo* synthesis of proteins and DNA has not been detected during repair from freeze damage.

Some of the many techniques developed to maximize the numbers of damaged cells recovered after freezing are presented in the following section. The substantial body of literature on this topic reflects the interest in ensuring that detection techniques are accurately determining the numbers of microorganims in food. The wide range of techniques developed attempts to address the varied mechanisms and sites of damage and the variety of conditions required for successful repair.

Many studies on freeze injury and repair have been conducted in model systems. Microorganisms that occur naturally in commercially frozen foods have received little study, and there is concern that microorganisms in naturally contaminated foods may have been subjected to a variety of stresses that are difficult to reproduce accurately in model systems (42, 93).

## V. DETECTION OF INJURED CELLS

The recovery and quantitative estimation of bacterial populations from foods that have been subjected to freezing injury require the addition of a resuscitation step prior to exposure to routinely used selective media. There is no universally used repair medium or protocol. Various workers have independently developed recovery media and methods for a range of target organisms. It is of interest to note that the food safety implications of freezing are not restricted to the recovery of pathogens from foods. Care needs to be taken in the way in which environmental samples such as swabs are handled, as it has been reported that the freezing and storage  $(-20^{\circ}\text{C})$  of sponges used for the sampling of beef carcasses significantly decreased recovery of *Salmonella* Typhimurium when present at low levels of less than or equal to 10 CFU cm<sup>-2</sup> (94).

#### A. Escherichia coli

Modified eosin methylene blue (MEMB), modified SD-39 (MSD), and modified sorbitol MacConkey agar (MSMA) were evaluated for their ability to recover *E. coli* O157:H7 from frozen (-20°C) high- and low-fat ground beef. MEMB and MSD proved more effective than MSMA. Recovery was better from high-fat ground beef. Since MSMA is a common selective medium for the detection and enumeration of *E.coli* O157:H7, the study highlighted the need for a more effective recovery medium (95). Recovery of freeze injured *E. coli* O157:H7 has been reported to be better on tryptic soy agar than modified eosin methylene blue agar, with MacConkey sorbitol and modified MacConkey sorbitol agars giving the lowest recovery (81).

In a collaborative study involving 20 laboratories, plating and immunological methods for the detection of *E. coli* in chilled and frozen samples of both ground beef and radish sprouts in combination with enrichment in modified *E. coli* broth supplemented

with novobiocin (mEC+n) were evaluated. Escherichia coli was readily recovered from frozen ground beef by all the plating methods studied with the exception of sorbitol MacConkey agar (SMAC). The sensitivity of all methods decreased when applied to radish sprouts and decreased further with sprouts that had been frozen. Based on this study, the authors recommended an immunomagnetic separation plating method using sorbitol MacConkey agar supplemented with cefixime and potassium tellurite (CT-SMAC) containing a beta-glucuronidase substrate in combination with static enrichment in mEC+n at  $42^{\circ}$ C (96).

When low levels of previously freeze-injured *E. coli* O157:H7 were inoculated into various foods containing higher levels of naturally competing microflora, recovery was reliably enhanced for most foods by allowing the food samples to stand at room temperature for 3 hours prior to selective enrichment with modified EC broth and novobiocin. For some highly acidic frozen foods (strawberries, vegetable juice) it was necessary to introduce a resuscitation step of 3 hours in a nonselective broth at room temperature prior to selective enrichment (97).

## B. Listeria monocytogenes

Several studies have demonstrated that the recovery of *L. monocytogenes* from foods can be enhanced by the careful selection of enrichment media (98, 82, 14, 93). A total of 549 samples of meat, fish, and poultry products purchased from retail outlets in Dublin, Ireland, were examined for the presence of *Listeria* species using a standard recovery method and a resuscitation method. The use of a standard recovery method involving direct plating on a selective medium, *Listeria* selective agar (Oxford formulation), found levels ranging from 0.7 to 5.0 log 10 CFU g<sup>-1</sup> on frozen product. The use of a recovery step involving solid-phase resuscitation on tryptone soya agar increased the numbers recovered by 2.5 log10 CFU g<sup>-1</sup>, indicating the presence of large numbers of injured cells (14).

The effectiveness of *Listeria* repair broth (LRB), buffered *Listeria* enrichment broth (BLEB), *Listeria* enrichment broth (LEB), Fraser broth (FB), and University of Vermont modified *Listeria* enrichment broth (UVM) in recovering and enumerating *Listeria* species from frozen fish fillets was assessed using a microwell plate method, the most probable number (MPN) technique. LEB and FB resulted in significantly lower MPNs than the three other media tested, with LRB recovering significantly higher numbers of *L. monocytogenes* cells than the other media tested (93). LRB as described by Flanders and Donnely (82) involves a 4 hour resuscitation step prior to the addition of selective agents.

#### C. Enterococci

The use of an overlay resuscitation method originally used by Ray and Speck (99) for the enumeration of freeze-injured enterococci on a selective medium was reported to enhance the recovery of enterococci from brain heart infusion (BHI) cultures stored at  $-18^{\circ}$ C for 1 week and thawed at 44°C. The resuscitation procedure involved surface plating on tryptic soy agar and incubating for 2 hours at 37°C, followed by overlaying the plates with 10 to 12 mL of selective enterococcus agar. The overlay procedure resulted in higher recovery of enterococci at both 37°C and 44°C and also helped reduce interference and false positive results from lactic acid bacteria also present in the system (100).

# D. Vibrio Species

Halophilic species have been reported to be particularly susceptible to stress due to heat or chilling and freezing during storage. V. parahaemolyticus and V. vulnificus were inoculated into a variety of seafood and cold stressed at  $2^{\circ}$ C to  $4^{\circ}$ C for 3 days and 7 days followed by freezing at  $-15^{\circ}$ C for 21 and 28 days. Akaline peptone water (APW) was found to be significantly more effective in recovering stressed cells than salt polymixin broth (SPB) (101).

#### VI. CYROPROTECTANTS

Cryoprotectants are solutes that protect all materials, including living cells, from freezing damage. Possible mechanisms of cryoprotection include the dilution of harmful solutes and the minimizing of the translocation of intracellular substances by strengthening the cell's membrane. Several naturally occurring food components, including sugars, amino acids, and peptides, and certain food additives, such as glycerol, are known to have a protective effect on microorganisms and therefore enhance survival during freezing and frozen storage. Some of these compounds may accumulate in the microenvironment of damaged cells as leakage products. If there is a large number of microorganisms present, the concentration of these compounds may become sufficient to provide protection.

Calcott and MacLeod (48) described an experiment in which *E. coli* survival was very low after rapid freezing in saline followed by slow thawing. The inclusion of 3% glycerol or 1% Tween 80 in the saline freezing medium resulted in nearly complete survival. The authors demonstrated that glycerol was able to reduce both damage to the cell wall and to the cell membrane, while Tween 80 prevented only membrane damage.

Glycerol added to phosphate buffer in the range of 2% to 4% was shown to protect *Listeria monocytogenes*, type Scott A, from injury during frozen storage at  $-18^{\circ}$ C and  $-198^{\circ}$ C for up to 6 months (64). It was further observed that 30 minutes of frozen storage with 2% glycerol was required before any protection was evident. In the short term (2 weeks or less), lactose, milk fat, and casein were superior to glycerol in maintaining the viability of *L. monocytogenes*. During long-term frozen storage, however, glycerol proved superior (64).

Since protectants such as glycerol and other polyols are frequently used to enhance sensory quality in a variety of frozen food products, it should be noted that these treatments may also affect product safety through the unintended protection of microbial pathogens.

#### VII. HYGIENIC PROCESSING OF FROZEN FOODS

Prepared and semiprepared frozen foods require extensive handling and processing prior to freezing, including size reduction measures such as cutting, dicing, and mincing, all of which increase the potential for microbial contamination because of the additional processing steps and the increase in product surface-to-volume ratios. In order to minimize microbial contamination and growth, the techniques used in handling and processing prior to freezing as well as control of temperature and the freezing rate are of critical importance.

The National Food Center in Dublin, Ireland, published the following summary recommendations for ensuring microbiological safety and sensory quality of both chilled and frozen foods (1):

Maintain high levels of hygiene at all stages of the product's life.

Chill and freeze products quickly and adequately after preparation and manufacture.

Brown (42) classified typical rates of freezing as follows: slow freezing: between 1°C and 10°C per hour; commercial freezing: between 10°C and 50°C per hour; rapid freezing: above 50°C per hour. Although rapid freezing has less effect on microbial cells, it results in less damage to the food and thus promotes better sensory qualities.

Rigidly maintain chill ( $< 5^{\circ}$ C) and frozen ( $< -18^{\circ}$ C) temperatures wherever possible during processing, storage, and distribution.

Ensure that chilled or frozen products are transferred in a continuous operation between adjacent temperature controlled areas, such as from delivery trucks to holding stores or from storage areas to retail display units. Transfer points are well known problem areas. A concept called the relay system, in which the food product is transferred safely from one responsible person to another with documented sign-offs is likely to enhance food quality and safety and significantly reduce risks.

Segregate cooked and uncooked chilled or frozen products in storage and retail display cabinets. For example, segregate uncooked meats and ready-to-eat meat products.

Conduct frequent and systematic temperature checks on chilled and frozen food products using appropriately calibrated instrumentation.

Do not overload chilled or frozen retail cabinets with products.

Train and educate all personnel (including consumers) in the correct handling and storage of frozen foods. Re-educate them when new practices are adopted.

The type of freezer and its configuration with respect to loading and placement of product can influence microbiological quality. The time required for the temperature of a food to decrease to below the minimum temperature for growth can vary considerably depending on the type of freezer used. For example, Eriksson and Löndahl (102) reported that the total bacteria counts on prefried meatballs packed in 5 kg cartons and placed on pallets with 50 mm spaces between the carton layers in a stationary freezing tunnel increased tenfold in the outermost layers of the pallet and hundredfold in the inner parts during the freezing process.

The cleanliness of freezing equipment itself has traditionally been considered of less importance than that of other equipment involved in the production of frozen foods. This was probably because it was assumed that the low temperature and the periodic removal of debris during defrosting would be adequate to prevent microbial growth. However, an increased understanding of the total process and rising concerns for food safety have resulted in more stringent requirements for food processing, including the freezing equipment employed.

Hygiene and cleanability need to be considered from the design stage on. Materials used in freezer construction should be durable, corrosion resistant, nontoxic, nonabsorbent, and capable of being easily cleaned and disinfected. As all surfaces need to be cleaned, equipment that comes in contact with food should be designed to avoid superfluous surface area. Horizontal surfaces should be avoided and replaced with sloping ones for simple drainage. Round profiles are better than square; sharp edges and sharp

corners should be avoided in favor of rounded edges. Welding, which gives smooth surfaces, is preferred to overlapping borders and the use of rivets or screws. All systems should have adequate drainage to avoid any pooling of water and debris or cleaning fluids (102).

The complete cleaning of freezing equipment generally requires four steps. The first step is the manual removal of product debris and snow prior to defrosting. In the second step, the freezer is defrosted and the walls, floors and other large surfaces are manually rinsed to remove the remaining debris. After the initial manual cleaning, automatic clean in place (CIP) systems can be used. These may involve the use of spray arms and nozzles to get the rinse water, detergents, and disinfectants to all parts of the freezing system while minimizing contamination to the plant as well as conserving water and chemicals. Particular attention needs to be given to the cleaning of belt stacks. After cleaning, the freezer should be dried using fans and/or a dehumidifier to limit the water available for microbiological growth. This last step is particularly important if the freezer is to be left sitting for several hours or over a weekend before the temperature is taken down and production is begun (102).

HACCP programs for food freezing operations are now in routine use throughout the world. The preemptive nature of such programs, which appears to enhance frozen food safety and quality, has been a boon to the industry. HACCP programs typically limit the use of microbiological testing to indirect purposes such as establishing limits for numbers of bacteria in new products or to verify existing food safety controls through end product sampling and challenge tests. The routine use of microbiological testing for direct control purposes is subject to practical limitations because the testing methods typically used cannot provide timely results. Microbiological testing may also be cost-prohibitive. As an alternative, quality control procedures based on the measurement of chemical or physical properties are generally faster and less expensive (1). Newer rapid microbiological methods such as bioluminescence (103) may in the future provide better opportunities for obtaining more direct and timely information on microbiological quality and safety.

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# 34

# **Frozen Food Plants: Safety and Inspection**

#### Y. H. Hui

Science Technology System, West Sacramento, California, U.S.A.

#### I. INTRODUCTION

The Departments of Agriculture of most states in this country have issued some basic regulations governing frozen food processing plants. Such regulations have, among others, the major objective of assuring that the frozen food products are safe for public consumption.

This chapter provides a modified version of those regulations issued by the Pennsylvania Department of Agriculture. The modification transforms a legal document into an easy-to-read scientific discussion. Consult the Pennsylvania Code for a copy of the original legal document (Title 7 Agriculture, Part III Bureau of Food Safety and Laboratory Services, Chapter 37, Frozen Foods, Subchapters B–G, Sections 37.11–37.216).

### A. Definitions

Accessible. Easily exposed for cleaning and inspection with the use of simple tools, such as those normally used by maintenance personnel.

Air temperature. The equilibrated temperature of the air environment in question.

*Breakup room.* Any area, or space within a warehouse, used primarily for the purpose of organizing cased frozen food into lots for individual consignment on route delivery.

Display case. Any case, cabinet or other facility used for displaying frozen food for sale.

Food product zone. Those surfaces with which food is normally in contact and those surfaces with which food may come in contact during processing, conveying, holding, refrigeration, and packing, and that may drain onto product contact surfaces or into the product.

Freezing cycle. Lowering the internal product temperature of a food product to a temperature of 0°F or lower.

*Frozen food.* Any article used for food or drink by man or other animals that is all of the following:

*Note*: Most data in this chapter have been modified with permission from documents prepared by Science Technology System, West Sacramento, California.

- *Processed.* Packaged and preserved by freezing in accordance with good commercial practices. Intended for sale in the frozen state.
- Internal product temperature. The equilibrated product temperature of a frozen food.
- Operator. (a) Any person, firm, or corporation operating or maintaining a frozen food plant or warehouse for the purpose of commercially preparing or storing frozen food. (b) Any person, firm, or corporation operating or offering to operate a vehicle for the purpose of transporting frozen food.
- Readily (or easily) accessible. Easily exposed without the use of tools, for cleaning and inspection.
- *Readily removable.* When a component part shall be capable of being separated from the principal part without the use of tools.
- Ready to eat frozen food. A frozen food product that has been factory processed to the point at which it is ready for use as a food and may or may not require further heating before use.
- *Removable.* When a component part shall be capable of being separated from the principal part with the use of simple tools, such as those normally used by maintenance personnel.
- *Retail outlet*. Any building, room, or parts thereof, where the sale of frozen food is conducted to the ultimate consuming purchaser.
- Route delivery. The transportation of frozen food, with frequent stops for partial unloading.
- Sale. Any and every transaction including the dispensing, giving, delivering, serving, exposing, storing, or any other possessing of frozen food wherein frozen food is subject to transfer to another person.
- Storage room or facility. Any area or space within a warehouse used for the purpose of storing frozen food.
- *Transportation*. The physical movement, or the acceptance for physical movement, of frozen food by a carrier.
- Vehicle. Any van, truck, trailer, automobile, wagon, ship, barge, freight car, airplane, or other means for transporting frozen food.
- Warehouse. Any structure, or room or part thereof, used for the purpose of storing commercially processed or manufactured frozen food.

#### **B.** Temperatures

#### 1. Air Temperature

All frozen food should be held at an air temperature of  $0^{\circ}F$  or lower, except for defrost cycles, for loading and unloading, or for other temporary conditions beyond the immediate control of the person under whose care or supervision the frozen food is held. Only those frozen foods destined for further processing or repackaging in smaller units should be defrosted for such purposes. All such defrosting should be in accordance with good sanitary precautions.

### 2. Internal Product Temperature

The internal product temperature of frozen food should be maintained at  $0^{\circ}F$  or lower except when the product is subjected to the conditions provided and relating to air temperature. When the frozen food is subjected to such conditions, the internal product

temperature should not exceed 10°F except during further processing. In all cases the product should be returned to 0°F as quickly as possible.

When an accurate determination of internal product temperature of any case of frozen food fails without having sacrificed the packaged frozen food, representative packages or units should be opened to allow insertion of the sensing element to the approximate center of the packages in question.

Internal product temperature of cases of consumer packages of frozen food should be determined in the following manner:

- 1. Open the top of the case and remove two corner packages.
- 2. Punch a hole in the case from the inside. The stem of the thermometer should not be used for punching. The hole should be positioned so that when the thermometer stem is inserted from the outside it fits snugly between packages.
- 3. The temperature may be read after 5 minutes.
- 4. Thermometers or other temperature measuring devices should have an accuracy of plus or minus 2°F.

#### II. GENERAL REQUIREMENTS

# A. Separation from Living Quarters and Objectionable Conditions

Frozen food preparation plants should be completely separated from areas used as living quarters by solid and impervious floors, walls, and ceilings, with no connecting openings.

Food processing plants should be located in areas reasonably free from objectionable odors, smoke, fly ash and dust, or other contamination. Objectionable conditions are often prevalent in the environs of the following facilities, though not limited to such facilities:

- 1. Oil refineries
- 2. City dumps
- 3. Chemical plants
- 4. Sewage treatment plants
- 5. Dye-works
- 6. Paper pulp mills

#### B. Accessways, Parking Areas, Expansion

Adequate dust-proof accessways for all vehicular traffic, connecting loading and unloading areas of the plant to the public streets, should be available. Employee parking areas and access roads close by the food processing plant should be hard surfaced with a binder of tar, cement, or asphalt.

When planning a plant, due consideration should be given to providing for an arrangement of buildings and necessary space to permit future expansion. Coolers, freezers, and the various processing departments should be so located that they may be enlarged without adversely affecting other departments.

# C. Potable Water, Nonpotable Water

The plant should have an ample supply of potable water available from an approved public or private source as specified.

Whenever a nonpotable water supply is necessary, it should not be used in a manner that will bring it into contact with the product or product zone of equipment. Nonpotable water systems should be kept entirely separate from the potable water supply. The nonpotable water lines should be positively identified by paint of a distinctive color.

# D. Equipment Installation, Hot and Cold Water, Cleanup, Sewage Systems

Equipment should be so installed and used that back siphonage of foreign liquids into the potable water lines is impossible.

Hot and cold water in ample supply should be provided for all plant cleanup needs. Hoses used for cleanup should be stored on racks or reels when not in use.

Disposal of liquid wastes should be through the public sewage system, if available and permitted by local ordinances, or by a properly designed and installed private facility. Private liquid waste treatment facilities should be approved by the health authority having jurisdiction.

#### III. PLANT LAYOUT

# A. Product Preparation and Processing, Preparatory Operations Areas

Product preparation and processing (including freezing) departments should be of sufficient size to permit the installation of all necessary equipment with ample space for plant operations, and with unobstructed truckways for conveyances of raw materials and processed products. The plant should be so arranged that there is a proper production flow of materials, without undue congestion or back-tracking, from the time raw materials are received until the frozen, packaged article is shipped from the plant.

Raw material storage rooms and areas where preparatory operations, such as washing and peeling of fruits and vegetables and the evisceration of poultry, are carried on should be separate from areas where frozen food is formulated, processed, and packaged. Doors connecting various rooms or openings to the outside should be tightly fitted and kept in a closed position by self-closing devices.

# B. Refrigeration Facilities, Quick Freezing Facilities, Waste Storage Rooms

Facilities for holding products under refrigeration until processed should be provided.

Whenever facilities for quick freezing of the processed product are used, they should be so located as to be convenient to the food processing and packaging departments. Ample freezer storage should be provided, located conveniently to the quick freezing facilities. Freezer storage should not, however, be required if the frozen products are immediately removed from the establishment.

Separate rooms for storing inedible materials such as fruit and vegetable peels and feathers and bones pending removal from the plant, should be provided in a location convenient to the various preparation and processing areas.

Waste storage rooms should be of sufficient size to permit the proper storage of filled and empty metal or other relatively nonabsorbent refuse containers and their lids. Waste storage rooms should be equipped with efficient power exhaust ventilation systems, hot and cold water outlets, and adequate floor drainage. The discharge from the exhaust system should be located well away from fresh air inlets into the plant.

# C. Storage of Packaging and Labeling Materials, Facilities for Inedible Products, Cleaning Room, Dockage Areas

Packaging and labeling materials should be stored in an area separate from but convenient to the packaging department, except that small quantities of such supplies necessary for maintaining continuity of operations may be stored in the processing and packaging departments.

Facilities for inedible products and catch basins should be located so as to avoid objectionable conditions affecting the preparation and handling of edible products.

A separate room or area and the proper facilities for cleaning equipment such as trays, hand trucks, and implements should be provided in a location convenient to the processing department. A power exhaust system should be provided to dispel steam and vapors from the room.

Dockage areas should be of adequate size, constructed of impervious materials, and so drained as to minimize the entrance into the plant of dust, dirt, and other contaminants from the receiving and shipping operations. If live animals are received, a separate dock should be provided for this purpose.

# D. Dressing Rooms, Toilet Rooms, Eating

Well-located, properly ventilated dressing rooms and toilet rooms of ample size should be provided for employeees. Dressing rooms should be separated from adjoining toilet rooms by tight, full height walls or partitions. Toilet rooms should not be entered directly from a work room but through an intervening dressing room or a properly ventilated toilet room vestibule.

Standard building codes should govern such matters as the following:

- 1. Ventilation and lighting of toilet and dressing rooms
- 2. Ratio of toilet, handwashing facilities, and urinals to the number of employees using such facilities
- 3. Type of fixtures used
- 4. Manner of installation of plumbing in such rooms

Employees should not eat in food processing or packaging areas.

# **IV. PLANT CONSTRUCTION**

# A. Floors, Walls, Ceilings, Window Ledges, Rodents, Vermin

Floors should be constructed of durable material that is easily cleaned and skid resistant. Where floors are wet cleaned, they should be sloped to drain.

#### B. Regarding the Walls of the Plant

1. Interior walls should be constructed of smooth, cleanable surfaces applied to a suitable base.

- 2. Dressed lumber should be used for exposed interior woodwork. Exposed wood surfaces should be finished with nontoxic oil or plastic paint or treated with hot linseed oil or clear wood sealer.
- 3. Coves with radii sufficient to promote sanitation should be installed at the juncture of floors and walls in all processing rooms.

Ceilings should be of adequate height and of smooth, cleanable material.

Window ledges should be sloped at least 45° to the interior to promote sanitation.

Frozen food plants and warehouses should be so constructed as to be rodent resistant. Exterior window and door openings should be equipped with effective insect and rodent screens. Where doors in outside walls of food handling areas are used for loading or unloading, fly chasers, fans, and ducts or other effective means should be provided at such doors to prevent the entrance of insects.

# C. Stairs, Refrigerator Doors, Variations from Requirements

Stairs in product handling departments should be constructed with solid treads and closed risers and should have side curbs of similar material, which should be 6 inches high as measured at the front edge of the tread.

Regarding the refrigerator doors,

- 1. Refrigerator doors and jambs should be covered with rust-resisting metal securely affixed to the doors and jambs.
- 2. Joints necessary for installation should be welded, soldered, or otherwise effectively sealed.
- 3. The juncture of the metal covering on jambs and walls should be sealed with a flexible sealing compound.
- 4. Doorways through which the product is transferred, either on overhead rails or on hand trucks, should be sufficiently wide to permit free passage of the largest trucks or the widest suspended products without contact with the jambs.

The requirements for building materials listed in this chapter represent minimum requirements. Variations should be acceptable provided the substitutions are equal to or exceed minimum requirements.

#### V. PLUMBING AND FLOOR DRAINAGE

# A. Wet Processing Areas, Hand Washing Facilities, Sterilizers

Floors should be sloped and drains functionally located to provide adequate drainage. In wet processing areas, the type and size of floor drains and sanitary sewage lines used and the method of installing such facilities and other plumbing equipment should conform to Commonwealth or local regulations.

Hand washing facilities should be located conveniently to all locations where products are prepared and processed. Lavatories should be supplied with the following:

- 1. Hot and cold or warm running water
- 2. Powdered or liquid soap in a suitable dispenser
- 3. An ample supply of single-service towels or electric air dryers
- 4. A suitable receptacle for used towels

Where sterilizers are required they should be large enough to allow complete immersion of tools and other implements. Sterilizing facilities should have the following:

- 1. A water line
- 2. A means of heating the water
- 3. An overflow outlet
- 4. A means of emptying the receptacle

# VI. LIGHTING; VENTILATION

# A. Work Rooms and Dressing Rooms, Fresh Air Intakes, General Light Intensities

#### 1. Work Rooms and Dressing Rooms

- 1. Work rooms and employee dressing rooms should have means for furnishing adequate natural light, which may be accomplished by having windows or skylights of an area approximately 25° that of the floor area.
- 2. Ventilation or efficient air conditioning or a mechanical ventilation system should be provided.
- 3. Adequate artificial light should be provided.

#### 2. Fresh Air Intakes

- 1. Fresh air intakes for mechanical ventilation systems should be equipped with effective replaceable filters to prevent the entrance of airborne contaminants.
- 2. Fresh air intakes should be located well away from power exhaust system discharges and other sources of airborne contaminants.

#### 3. General Light Intensities

- 1. The general light intensities in product preparation, processing, and packaging areas should be not less than 20 foot-candles as measured 30 inches above the floor.
- 2. Where detailed visual tasks are required to assure a safe, wholesome product, the intensity of light on the surfaces of the product or product container should be not less than 50 foot-candles.
- 3. At least ten foot-candles of light should be provided in all dressing rooms and at least 5 foot-candles in all other areas of the plant.

#### VII. FROZEN FOOD PROCESSING EQUIPMENT: CLASSIFICATION

These specifications should apply to the design, materials, construction, and installation of equipment used in the processing, holding, and packaging of ready-to-eat frozen food and the processing and holding of gravies, coating batters, and other food ingredients containing eggs, milk, broth, and other food components capable of supporting rapid bacterial growth.

Design, materials, construction, and installation of frozen food equipment should be easily accessible for cleaning and sanitizing.

In order to encourage the cleaning of equipment, the time factor and the ease of disassembly are important considerations. The unit of equipment should contain the fewest number of parts to permit easy reassembly by unskilled labor following cleaning.

# A. Group A

Equipment in Group A should be used for the processing, conveying, holding, refrigeration, and packaging of gravies, coating batters, or other food ingredients containing eggs, milk, or broth, alone or in combination with other food ingredients, that are capable of supporting rapid bacterial growth. This group includes, but is not limited to, the following:

- 1. Pumps
- 2. Valves
- 3. Pipelines and fittings
- 4. Heat exchangers
- 5. Homogenizers
- 6. Containers
- 7. Hoppers
- 8. Fillers

# B. Group B

Equipment in Group B should be used in the processing, holding, and conveying of foods or food ingredients that are intended to be incorporated in ready-to-eat frozen food. This group includes, but is not limited to, the following:

- 1. Reservoirs
- 2. Holding tanks
- 3. Kettles
- 4. Mixers for liquids
- 5. Mixers and blenders for powders
- 6. Dough mixers
- 7. Flour handling equipment
- 8. Fryers
- 9. Cutters
- 10. Dicers
- 11. Slicers
- 12. Cutting boards
- 13. Pumps
- 14. Valves
- 15. Tanks
- 16. Lines and fittings for liquid sugar
- 17. Lines and fittings for oil and shortening

# C. Group C

Equipment in Group C should be used in the manufacture of ready-to-eat frozen food, but applicable standards are not available.

# VIII. FROZEN FOOD PROCESSING EQUIPMENT: MATERIALS, DESIGN, AND CONSTRUCTION: GROUPS A AND B

Specifications and published standards for food equipment have been developed by official agencies and voluntary organizations other than those specifically mentioned in this chapter. These standards may be worthy of consideration in the evaluation of certain equipment items. The development organization and the area in which standards are published are the following:

- 1. National Sanitation Foundation, Food preparation and service equipment
- 2. United States Department of Agriculture, Meat Inspection Division, Meat processing equipment
- 3. United States Department of Agriculture, Poultry Inspection Division, Poultry processing equipment
- 4. United States Department of Commerce, National Marine Fisheries Service, Fishery products handling and processing equipment

# A. Group A

Effort should be made to have equipment in Group A conform to 3A Sanitary Standards. Standards are as follows:

- 1. Pumps. 3A Sanitary Standards for Pumps for Milk and Milk Products, Including Both Centrifugal and Rotary Pumps
- 2. Valves. 3A Sanitary Standards for Inlet and Outlet Leak Protector Plug Valves for Batch Pasteurizers
- 3. Milk and milk products equipment. 3A Sanitary Standards for Fittings and Connections Used on Milk and Milk Products Equipment
- 4. Heat exchangers. 3A Sanitary Standards of Plate Type Heat Exchangers for Milk and Milk Products
- 5. Pasteurizers. 3A Accepted Practices for the Sanitary Construction, Installation, Testing, and Operation of High-Temperature, Short-Time Pasteurizers

#### B. Group B

Effort should be made to have equipment in this group conform to B.I.S.S.C. standards, which are promulgated by the Baking Industry Sanitation Standards Committee. Standards (always check for the latest version) are as follows:

- 1. Mixers or blenders for powders. B.I.S.S.C. Standards pending
- 2. Horizontal and vertical dough mixers. B.I.S.S.C. Sanitary Standard No. 6, for Horizontal Mixers and Vertical Mixers
- 3. Flour handling equipment. B.I.S.S.C. Sanitation Standard for Flour Handling Equipment
- 4. Liquid sugar handling equipment. B.I.S.S.C.
- 5. Liquid oil and shortening handling equipment. B.I.S.S.C.
- 6. Fryers. B.I.S.S.C. Sanitation Standard No. 16, for Doughnut Equipment
- 7. Depositors, fillers. B.I.S.S.C. Sanitation Standard No. 5, for Cake Depositors, Fillers and Icing Machines
- 8. Conveyors. B.I.S.S.C. Sanitation Standard No. 7, for Conveyors

9. Homogenizers, emulsifiers. B.I.S.S.C. Sanitation Standard No. 18, for Emulsifiers and Homogenizers

# IX. FROZEN FOOD PROCESSING EQUIPMENT: MATERIALS, DESIGN, AND CONSTRUCTION: GROUP C

#### A. Materials

#### 1. Food surfaces

- (a) Surfaces within the food product zone should be smooth, free from pits, crevices, and loose scale, and relatively nonabsorbent. Furthermore, surfaces should be nontoxic and unaffected by food products and cleaning compounds.
- (b) Sponge rubber, stone slab, linoleum, flannel, and unglazed ceramic material are basically objectionable and should not be used.
- (c) Wood and cloth, if used, should be indicated under specific application.
- 2. The finish of corrosion-resistant surfaces such as stainless steel or nickel alloy should be of 125 grit, and properly applied.
- 3. Finishes of cast iron, cast and forged steel, and cast nickel alloy should not exceed a surface roughness of American Standard #125 or its equivalent.
- 4. The use of galvanized surfaces should be minimal and where used should be of the smoothness of high quality commercial hot dip.
- 5. Copper and its alloys should not be used in equipment where edible oils, liquid shortening, chocolate liquor, or other fatty food products come in contact with the metal.
  - 6. Cadmium should not be used in any manner or form on the food equipment.
- 7. Lead should not be used within or adjacent to the food product zone, with the exception of its inclusion in dairy solder, in an amount not to exceed current specification.
  - 8. Plastics should be in conformity with federal regulations.
- 9. Gasketing and packing materials should be relatively nonporous, relatively nonabsorbent, and installed in a manner that results in a true fit to prevent protruding into the product zone of the creation of recesses or ledges between the gasketed joints.
- 10. Coatings used in the food product zone as a lining to prevent corrosion of the base material of food equipment should be in conformity with federal regulations.

# B. Design and Construction in the Food Product Zone

- 1. All parts of the product zone should be readily accessible or should be readily removable for cleaning and inspection.
- 2. All parts of the food product zone should be free of recesses, dead ends, open seams and gaps, crevices, protruding ledges, inside threads, inside shoulders and bolts or rivets that form pockets and patterns.
- 3. Permanent joints of metal parts should be butt welded. Dissimilar metals should not be used in equipment construction if their contact with liquid products might create deleterious chemical or electrolytic action.
- 4. Welding within the food product zone should be continuous, smooth, even, and flush with the adjacent surfaces.
- 5. Interior corners should be provided with a minimum radius of one quarter inch except where a greater radius is required to facilitate drainage or cleaning.

- 6. Equipment should be constructed and installed to provide sufficient pitch so as to be completely self-draining.
- 7. Equipment that introduces air into the food product or uses air to convey the food product should be fitted with filters capable of withholding particles 50 microns or larger in size. Such filters should be readily removable for cartridge replacement or cleaning.
- 8. Bearings should be located outside the food product zone or outboard, and should be of the sealed or self-lubricated type. Those intended for use with a dry granular or a dry pulverized product directly adjacent to the food product zone should be of the sealed type without grease fittings. The bearings should be installed flush to eliminate any recessed areas around the shaft within the food product zone.
- 9. Shaft seal assemblies and packing glands should be outboard and should be readily removable. The shaft seal or packing should be retractable within a space between the assembly and bearing to facilitate easy removal of the sealing assembly and materials for cleaning and inspection.
  - 10. Permanent screening and straining devices and surfaces:
  - (a) All permanent screening and straining devices should be readily removable for cleaning and inspection. They should be designed to prevent replacement in an improper position.
  - (b) Permanent screening and straining surfaces intended for use with a liquid or a semiliquid product should be fabricated from perforated metal.
  - (c) Permanent screening and straining surfaces for use with a dry granular or a dry pulverized product should be designed with sufficient strength for their intended use and be sized to remove foreign material efficiently.
  - 11. Filtering process:
  - (a) Filtering surfaces should be readily removable for cleaning and inspection.
  - (b) Filter papers should be of the single-service type.
  - (c) Filter cloths and spun glass filters should be launderable.
  - 12. Hinges and latches should be of the simple take-apart type.
- 13. Motors should be of the totally enclosed finless type and should be mounted on the equipment whenever possible.
- 14. Covers should be provided on reservoirs, hoppers, or other vessels and should be readily removable and fitted with drip protective devices or facilities to prevent foreign substances from falling into the product.

# C. Design and Construction in the Nonfood Products Zone

- 1. Safety and gear guards should be removable for cleaning and inspection.
- 2. External surfaces should be free of open seams, gaps, crevices, unused holes, and inaccessible recesses.
- 3. Horizontal ledges and frame members should be kept to a minimum. External angles should be rounded, and internal angles should be avoided.
- 4. Where lubrication of equipment is required, provision should be made to prevent leaking or dripping into the food product zone.

#### 1. Installation of equipment

- 1. Equipment should be installed on a foundation of durable, easily cleaned material.
- 2. Equipment should be placed at adequate distance from walls, ceilings, and floors for cleaning and maintenance, or sealed watertight thereto. The preferred minimum space between walls or ceilings should be 30 inches.
- 3. Whenever equipment passes through walls or floors it should be sealed to that partition, or sufficient clearance should be allowed to permit inspection, cleaning, and maintenance.
- 4. Wherever there is spill or drip, drains and catch pans should be provided and should be of such dimensions as to collect all spill and drip. They should be easily accessible or easily removable for cleaning.
- 5. Where pipes pass through ceilings of processing areas, pipe sleeves should be inserted in the floor above so that their upper periphery is at least two inches above the floor.

#### 2. Connections

All electrical connections, such as switch boxes, control boxes, conduits, and box cables, should be installed a minimum of  $\frac{3}{4}$  inch away from the equipment or walls or be completely sealed to the equipment or walls.

# X. OPERATING PRACTICES FOR THE COMMERCIAL MANUFACTURE OF FOOD

#### A. Handling and Storage of Materials

# 1. Requirements for Food Should Be as Follows:

- 1. All food ingredients received at the plant should be wholesome.
- 2. Storage conditions should protect against contamination from rodents, insects, and other sources.
- 3. Storage temperature should be in accordance with the following practices:
  - (a) Ingredients requiring refrigeration should be stored at an air temperature of 40°F or lower. Only areas where the temperature does not exceed 40°F should be considered refrigerated.
  - (b) Frozen ingredients not in process should be stored at an air temperature of  $0^{\circ}F$  or lower.

Storage of packaging materials should be separate and set apart from food preparation and processing operations under conditions which should protect against contamination from rodents, insects, and other sources.

General housekeeping should be conducted so that the plant and premises present a neat and orderly appearance at all times.

#### B. Personnel Hygiene

The services of an employeee with any open sore on an exposed portion of the body or one afflicted with an infectious or contagious disease should not be used except that services of employees with finger cuts or with bandaged finger cuts and similar type coverings may be

utilized on the condition that the employeee wear rubber gloves. Any employeee with an upper respiratory infection should be assigned duties outside of the areas of food preparation, processing, and packaging. Visitors to food preparation, processing, and packaging areas should comply with employee requirements.

Practices for employees handling unpackaged food should be as follows:

- 1. Employees should wear head covering and should keep clothing in a clean condition consistent with the duty being performed.
- 2. Before beginning work, after each absence from post of duty and after contact with nonsanitized surfaces, each employee should
  - (a) Wash his hands with liquid or powdered soap and warm water dispensed from a foot or elbow operated device (existing faucet facilities need be changed to a foot or elbow operated device only when a new hand washing facility is installed).
  - (b) Rinse his hands in a chlorinated spray or other approved sanitizing agent, unless a bacteriostatic soap is used in washing.
  - (c) Dry his hands with single-service towels or with electric hot air dryers.
  - 3. Hand contact with food products should be minimized.
  - 4. Use of a common dip bowl or tank is prohibited.
- 5. Whenever rubber gloves are used they should be cleaned and sanitized in accordance with hand washing specifications.
- 6. Use of tobacco in any form, chewing gum, or eating in rooms where food products are stored, handled, or prepared should not be permitted.

# C. Plant and Equipment Sanitation

- 1. Plant and equipment should be clean when put into service.
- 2. All floors, tables, splash boards, work surfaces, equipment, and utensils should be maintained in a clean and sanitary condition at all times. Critical areas and all food contact surfaces should be cleaned and sanitized whenever necessary or at scheduled intervals.
- 3. Equipment such as pipes, pumps, fillers, and valves should be dismantled for cleaning and sanitizing, unless in-place cleaning and sanitizing methods are effective. Suggested criteria for acceptance of clean-in-place (C.I.P.) systems areas are as follows:
  - (a) The arrangement should allow cleaning and bactericidal solutions to be circulated through the system.
  - (b) Solutions should touch all surfaces.
  - (c) The system should be self-draining or otherwise completely evacuable.
  - (d) The cleaning procedure should result in thorough cleaning of the equipment.
- 5. A thorough rinse with potable water should follow any sanitizing operation that has been completed with a chemical sanitizing agent.

#### D. Preparation and Processing

1. Fans, blowers or air cooling systems should not move unfiltered air from raw material or preparation rooms into processing rooms.

- 2. Only adequately cleaned, prepared raw materials should be introduced into areas where frozen precooked foods are cooked and subsequently handled in processing operations.
- 3. Preparatory operations feeding to the packing line should be so timed as to permit efficient handling of consecutive packages in production, and under conditions designed to prevent contamination, loss of quality, and spoilage.
- 4. When batter, egg wash, or milk wash is an ingredient, it should be maintained at a product temperature not to exceed 45°F, except when the process temperatures required for manufacturing the product are higher. Cracked or flaked ice used to refrigerate batters should meet bacterial standards for potable water. Batter remaining in machines and equipment at cleanup time should be discarded.
- 5. Breading materials that have come in contact with batter and have been removed by screening should be discarded.
- 6. Food ingredients or mixtures that are capable of supporting rapid bacterial growth should be maintained either at a product temperature above 160°F or below 45°F, except when processing temperatures falling in this range are an integral part of the product manufactured, such as yeast.
- 7. Cooked food such as meat, poultry, sauces, and gravies should be all of the following:
  - (a) Refrigerated or incorporated into the finished product within 1 hour following preparation.
  - (b) Refrigerated within 30 minutes following preparation at an air temperature of 50°F or less if the product is to be held from 1 to 8 hours after preparation.
  - (c) Refrigerated within 30 minutes following preparation so that the internal temperature of the food product will be 40°F or lower, within 2 hours of refrigeration if the food product has been comminuted, sliced, or is a liquid, and if the food is to be held more than 8 hours. Large solid food components such as those that must be cooled before slicing should be refrigerated at an air temperature of 40°F or lower.
- 8. Trays, pans, or other containers of ingredients destined for incorporation into the finished product should be protected with a clean cover unless these ingredients are used within 30 minutes of preparation. The cover should not be made from porous material.
- 9. Permanently legible code marks should be placed on each immediate container or package at time of packing. The code marks, as devised by management, should include date of packing and establishment where packed.
- 10. Packaged products should be placed in the freezer according to good commercial practice. Placement of packages in cases before freezing is prohibited unless the wholesome quality of the product is fully protected by prior processing.
  - 11. Waste disposal:
    - (a) Refuse from the food operations should be promptly placed in containers that are prominently marked REFUSE and equipped with lids.
    - (b) The handling of refuse should be done in such a manner as not to cause a nuisance.
    - (c) All refuse should be removed from the premises on a daily basis and in such a manner as not to contaminate food products being manufactured within the plant.

(d) Refuse containers should be thoroughly cleaned immediately after each emptying.

# E. In-Plant Freezing

- 1. During the freezing cycle products should be cooled to 50°F or lower within 2 hours.
  - 2. Products should then be reduced to 0°F. by approved commercial practice.
- 3. When necessary, products should be protected so that dehydration and discoloration will not occur during the freezing cycle.
- 4. The freezer should be precooled to an air temperature of  $0^{\circ}F$  before loading. During loading, however, the freezer may rise to temperatures above  $0^{\circ}F$  for short periods of time.
- 5. If cold air is used as the freezing medium the product should be arranged by staggering the individual items or by employing dunnage, spacers, or other suitable methods to permit satisfactory circulation of cold air around the products. The cold air should be circulated by a positive method; natural air circulation is not satisfactory.
- 6. The freezer and associated equipment used for handling the product should be maintained in a clean and sanitary condition at all times.
- 7. A suitable indicating or recording instrument should be used to measure the temperature of the cooling medium, that is, air, liquid, refrigerated plates, or pipe coils.
- 8. Packaged items should be frozen in a manner that will result in a minimum amount of bulging or distortion.
- 9. After the freezing cycle the frozen product should be transferred to a storage facility as quickly as possible.

#### XI. TRANSPORTATION EQUIPMENT

Vehicles used for transportation should be equipped with insulation and mechanical refrigeration systems, or other refrigeration methods or facilities capable of maintaining an air and product temperature of 0°F, or lower, while loaded with frozen food.

Vehicles used for transportation should be equipped with a thermometer or other appropriate means of temperature measurement, indicating air temperature inside the vehicle. The dial or reading element of the thermometer should be mounted on the outside of the vehicle.

Vehicles used for route delivery should comply with all equipment provisions specified in this chapter for vehicles used for transportation, and should in addition be equipped with curtains or flaps in the doorway area, or with port doors, or with portable insulated chests to maintain required temperature during distribution.

#### XII. HANDLING PRACTICES FOR OVER-THE-ROAD TRANSPORTATION

Vehicles should be precooled to an air temperature of 20°F or lower before loading.

Frozen food shipments should not be accepted for transportation when the internal product temperature exceeds 0°F, except that shipments in transit at a higher temperature should not be considered in violation of this section if the bill of lading, signed by the shipper, specifies that the product is consigned to a warehouse or other facility for further

freezing, or if the product is to be sold as fresh and is to be defrosted when offered for use or for sale.

Frozen food should be loaded in a transportation vehicle so as to provide free circulation of refrigerated air at the front, rear, top, bottom and both sides of the load, except for vehicles of envelope construction in which refrigerated air circulates within the walls of the vehicles.

The mechanical refrigerating unit of vehicles should be turned on and doors of vehicles should be kept closed or curtained during any time interval when loading or unloading operations cease.

The average product temperature of any shipment of frozen food should be determined during loading and unloading by adequate temperature readings.

#### XIII. HANDLING PRACTICES FOR ROUTE DELIVERY

Lots for individual consignment which are to be sold in a frozen state should be refrigerated by means of mechanical refrigeration, dry ice, or by any other means capable of maintaining an air and product temperature of  $0^{\circ}F$  or lower.

Insulated containers should be precooled to a temperature of 20°F or lower before being loaded with frozen food.

Doors of vehicles should be kept closed during any time interval that loading or unloading operations cease.

#### XIV. SANITARY REQUIREMENTS DURING TRANSPORTATION

Interior surfaces of vehicles and devices used for transporting frozen food should be clean and free of objectionable odors before being loaded with frozen food.

Frozen food should be securely packaged or wrapped in a sanitary manner before it is accepted for transportation.

#### XV. WAREHOUSING EQUIPMENT

Regarding refrigeration capacity and minimum temperature:

- 1. Warehouses should be equipped with suitable mechanical refrigeration capacity to maintain, under extreme outside temperature and peak load conditions, an air temperature of  $0^{\circ}F$  or lower.
- 2. Storage rooms and all their parts should be maintained at an air temperature of 0°F or lower.

Regarding the use of thermometers:

- 1. Each storage room should be equipped with a thermometer or some other temperature measuring device which is easily visible.
- 2. The sensing element of thermometers and other temperature measuring and recording devices should be located not more than 6 feet nor less than 5 feet from the floor and not in a direct blast of refrigerated air or near entrance doors.
- 3. When indicating thermometers alone are used they should be read and recorded at least once every 24 hours during each calendar day.

- 4. Recording thermometers equipped with charts should have a range of at least  $15^{\circ}$  above and  $10^{\circ}$  below,  $0^{\circ}$ F in graduations of  $1^{\circ}$ .
- 5. The use of electric or hand-wound clocks as well as 24-hour or 7-day charts for recording thermometers should be optional at the discretion of the operator.
- 6. Each chart or record of observed temperatures should be dated to show the time interval covered and should be kept on file for at least 1 calendar year.

Breakup rooms should be maintained at temperatures not in excess of 20°F.

#### XVI. WAREHOUSING HANDLING PRACTICES

The operator of a warehouse should not accept custody of a lot or shipment of frozen food if internal product temperature exceeds 0°F (except as relating to air temperature; and internal product temperature). When frozen food is accepted pursuant to such exception the operator should make a written record of the incident.

Notwithstanding the above, custody of lots with an internal product temperature not in excess of 10°F may be accepted by the operator on request of the owner of the lot in question if the foods are detained from sale at retail and the temperature of such product is promptly returned to and maintained at 0°F or lower.

Before lots of frozen food are placed in storage they should be given lot numbers for effective identification.

Regarding the storage of frozen food,

- 1. Frozen food in storage should be placed on dunnage, pallets, racks, or skids and should be stored no closer than 18 inches to the ceiling and otherwise stored so as to permit free circulation of refrigerated air.
- 2. Frozen food should be stored under good sanitary conditions that preclude injury and contamination from or to other food held within the warehouse.

During the defrosting of overhead coils in storage rooms stacks of frozen food should be effectively protected from contamination by condensation, drip, or leakage.

Breakup rooms should not be used for storage unless the temperature is kept below  $0^{\circ}F$ .

At time of removal from warehouse custody the internal product temperature of frozen food should not exceed  $0^{\circ}F$  unless authorized by the owner to begin a defrost cycle.

#### XVII. WAREHOUSING SANITARY PRACTICES

Floors, walls, and ceilings of a warehouse should be maintained in a good sanitary condition. Premises of a warehouse should be maintained in a good sanitary condition.

Warehouses should have water flush toilets so located as to be convenient to all employees. Toilet rooms should be well lighted and ventilated and should be maintained in a sanitary condition. The doors of all toilet rooms should be full-length and self-closing.

Adequate hand washing facilities, including hot and cold or warm running water, powdered or liquid soap in a suitable dispenser, and single service towels or hot air dryers, should be provided adjacent to all toilet rooms. Wash rooms should be well lighted and ventilated, and should be maintained in a sanitary condition. The use of a common towel is prohibited.

Warehouses should have a dressing room or rooms for the changing and hanging of wearing apparel. If individual lockers are provided, they should be well vented and maintained in a clean, sanitary condition, and should be free from disagreeable odors. The dressing room or rooms should be adequately lighted and ventilated and should be maintained in a clean, sanitary condition.

#### XVIII. RETAIL EQUIPMENT

Storage facilities should be equipped with suitable mechanical refrigeration capacity to maintain, under extreme outside temperature and peak load conditions, an air temperature of  $0^{\circ}F$  or lower.

When storage facilities of cabinet type are used they should be all of the following:

- 1. Defrosted as frequently as necessary to maintain refrigeration efficiently as specified
- 2. Equipped with a thermometer indicating a representative air temperature

When storage facilities of walk-in freezer type are used, the following requirements should apply:

- 1. Frozen food in storage should be on dunnage, pallets, racks, or skids and should be stored so as to permit free circulation of refrigerated air.
- 2. The facility should be equipped with a thermometer, the sensing element of which should be located within the upper third of the distance between floor and ceiling. The sensing elements should not be placed in a direct blast of air from cooling units, cooling coils, and heat exchange devices, or near the entrance door.
- 3. The facility should be equipped with an automatic mechanism for defrosting refrigerated coils when forced air blower refrigeration is used.

Frozen food display cases should be designed, constructed, and equipped with mechanical refrigeration facilities capable of maintaining an air temperature of 0°F or lower.

Frost on refrigerator coils and in air passages of display cases should be removed as frequently as necessary to maintain refrigeration efficiency of 0°F or below.

Each display case should be equipped with a thermometer, the sensing element of which should be located in an appropriate place within the path of refrigerated air being returned to the coils.

The product load line should be designated by a distinctive line at inside terminal ends of each display case, and such lines should be at the highest point of discharge and return of refrigerated air.

Separators in display cases should be located a minimum of one-half inch from terminal ends to provide for free circulation of refrigerated air between the terminal ends and the displayed product.

Display cases in retail outlets should be so placed as to be relatively free of all of the following:

- 1. Air current resulting from door drafts, electric fans, and other factors that adversely deflect the current of refrigerated air within the display case.
- 2. Heat elements such as lights, heating units, and related devices that tend to raise the temperature of refrigerated air within the display case.

#### XIX. RETAIL HANDLING PRACTICES

Frozen food should not be accepted for delivery by a retail outlet when the internal product temperature exceeds 0°F, except as relating to air temperature and internal product temperature. When frozen food is accepted under this exception, the retail outlet should duly record that fact, preferably on the bill of lading or the delivery ticket.

The receiving of frozen foods should follow the following recommendations:

- 1. All frozen food received at a retail outlet should be placed in storage facilities without undue delay.
- 2. Retail outlets should employ the first-in, first-out basis of inventory.

Retail outlets should be equipped with storage facilities of sufficient cubic displacement to accommodate the storage of frozen food.

Regarding storage and display:

- 1. Frozen food should not be placed above the product food lines within any display case.
- 2. Frozen food in retail outlets should be stored and displayed under good sanitary conditions.

Obligations for compliance with this retail requirement should cease at the time of retail sale or when the ultimate purchaser takes custody of the product.

A product should be understood to be in the custody of the purchaser when some one else takes a delivery from a retail outlet at the request of the purchaser.

#### A. Preordered Frozen Foods

Frozen food that is preordered by the ultimate consumer may be sold at internal temperatures exceeding 0°F but not exceeding 45°F provided all of the following are met:

- 1. A pickup time and date is announced to potential customers.
- 2. Potential customers are advised on the sales invoice that food is subject to possible quality and perishable degradation when internal product temperature exceeds 0°F and the products are not to be resold.
- 3. Frozen foods are limited to foods that do not support the rapid and progressive growth of pathogenic microorganisms.
- 4. Foods are not to be resold to other parties.
- 5. Frozen foods arrive at the pickup location with an internal temperature of 0°F or below except as relating to minimum temperature requirements.

#### XX. FROZEN PRECOOKED FOODS: ESTABLISHMENT INSPECTION

The United States Food and Drug Administration has issued the following guides regarding the inspection of establishing manufacturing frozen precooked foods.

- 1. Check for presence of rodents, birds, insects, or for other possible sources of contamination.
- 2. Check what tests are conducted on incoming raw materials (e.g., filth, mold, decomposition, bacterial load check), whether they are received under a salmonella-free guarantee or tested for salmonella and other pathogens.

- 3. Determine the adequacy of cleaning and sanitizing steps for equipment.
- 4. Watch for time-temperature abuses in processing, particularly where product may be hung up in equipment or in cooling and storage processes.
- 5. Be sure that cooking, cooling, and storage conditions are adequate and do not merely produce incubation temperatures for bacteria.
- 6. Consider employee hygiene and sanitary practices, mainly hand-washing and sanitizing, but including health, wounds, sores, or disease conditions.
- 7. Check food and color additives used to ascertain if permitted and used at proper levels
- 8. Check labeling and net weights for compliance.

During a comprehensive inspection of a frozen precooked foods establishment, cover

#### A. Raw Materials

- 1. Examine raw materials in storage for evidence of contamination with filth (insects, rodents, birds, etc.), mold, and possible routes of other microbiological contamination.
- 2. Determine if raw materials are stored and handled properly. (i.e., frozen products kept frozen, etc.).
- 3. Examine raw material area for possible misuse of pesticides, rodenticides, and other dangerous chemicals.
- 4. Determine if critical raw materials, e.g., NFDM, frozen eggs, dried eggs, are received under a salmonella-free guarantee and are tested for salmonella and other pathogens.
- 5. Check food and color additives in storage and determine if they are allowed for use.

# **B.** Processing

- 1. List product flow in detail, including a flow plan.
- 2. Ascertain if manufacturing equipment is suitable for its intended use and is in good state of repair.
- 3. Check if equipment is cleaned and sanitized properly before use, as necessary during the day (i.e., at breaks, etc.) and after use.
- 4. Obtain manufacturing process in detail including the conditions under which products are held prior to process, handled during process, and handled after process.
- 5. If applicable to the process, determine the time–temperature parameters of the manufacturing operation. Include
  - (a) Temperature of raw materials prior to process
  - (b) Temperatures during the process
  - (c) Time-temperature during and holding periods between steps
  - (d) Time-temperature of final heat treatment
  - (e) Temperature of product at final packaging
- 6. Check for any undue delays between final heat treatment and packaging.
- 7. Determine freezing process to include
  - (a) Freezing equipment and process

- (b) Holding time and temperature prior to freezing
- (c) Time taken to reach hard frozen condition
- 9. Check finished product handling and storage.
- 10. Evaluate the use of food and color additives to ascertain if permitted and used at the proper level.
- 11. Check use of pesticides and rodenticides to preclude their becoming incidental food additives.

#### C. Sanitation

- 1. Evaluate the firm's operation for compliance with GMPRs (Sanitation).
- 2. Check employee practices that could lead to the contamination of the products with filth, bacteria, and/or mold.
- 3. Determine if employees use hand dip and sanitizing solutions when necessary.

# D. Economics

- 1. Check the firm's net weights to ascertain proper container fill.
- 2. Review labeling for compliance with FPLA, etc.
- 3. Obtain significance of firm's coding system.

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# Frozen Dessert Processing: Quality, Safety, and Risk Analysis

### Y. H. Hui

Science Technology System, West Sacramento, California, U.S.A.

# I. INTRODUCTION

In 1989, the U.S. Food and Drug Administration (FDA) issued a document entitled Frozen Dessert Processing Guidelines. This document provides recommendations to make an assessment of the safety and quality of frozen desserts from raw ingredients to packaged finished products and sets action priority recommendations.

This chapter updates the information and discusses some general aspects of risk analysis and product safety for frozen desserts.

#### II. GENERAL INSTRUCTIONS

These guidelines provide recommendations to make an assessment of the safety and quality of frozen desserts from raw ingredients to packaged finished products and set action priority recommendations.

An action priority provides guidance as to what to do first in responding to product safety problems.

# Risk Assessment

Throughout these processing guidelines, each item or area has been assigned a suggested risk assessment: (H) *High Risk*, (M) *Moderate Risk* or (L) *Low Risk*. These are *suggested* risk assessments. These are general assessments and may not represent specific *individual* circumstances. If an observed condition constitutes a risk higher or lower than that suggested in these guidelines, the corresponding Action Priority would apply.

# **IMPORTANT**

The Risk is automatically "H" or "High" when the problem observed is a critical processing element involving

- 1. Proper pasteurization, whereby every particle of milk, milk product, or mix may not have been heated to the proper temperature and held for the required time in properly designed and operated equipment.
- 2. A cross-connection exists whereby direct contamination of milk, milk products, or mix is occurring.
- 3. Conditions exist whereby direct contamination of pasteurized product is occurring.

#### The Action Priorities

The three risk categories are defined in terms of appropriate monitoring levels and action priority:

(H) High Risk:

High level of control needed because of immediate impact on product safety. Potential for a problem is high without appropriate monitoring.

Action Priority—No product should be processed until the problem is corrected. Product on hand should be checked for contamination if appropriate. If product on hand is found to be contaminated, appropriate action should be taken.

(M) Moderate Risk:

Potential for a problem is somewhat limited to abuse or particular criteria. Timely monitoring is required because problems in these areas could result in a risk to product safety.

Action Priority—Correction of these problems is necessary within a short period of time. A few days or weeks may be reasonable. Specific additional monitoring is needed until the correction has been accomplished.

(L) Low Risk:

Monitoring needed only on inspection or random-checking basis. Risk potential is low, and significant risk would only result from extensive abuse or extenuating circumstances.

Action Priority—Correction is necessary to help assure ultimate product safety. However, the time frame for correction can be flexible and based around non-public-health issues such as production schedules. Until the correction is accomplished, routine checks should be made to provide assurance that the status has not changed to "M" or "H."

The action priorities in these guidelines were formulated to be compatible with a Hazard Analysis Critical Control Point (HACCP) system. However, in order to implement a full HACCP program, individual in-plant monitoring points and frequencies should be established.

#### III. INCOMING MATERIALS

Effective plant product safety requires control from the earliest stages of production. An integral part of a product safety program concerns those materials that are brought into the plant from the outside.

To be successful, an incoming materials program should address at least three concerns about each material:

- 1. The materials should come from an acceptable source.
- 2. The conditions of incoming product should be evaluated and found acceptable.
- 3. If established specifications are not met, then actions to be taken should be clearly provided for.

# A. Ingredients

Sources

Risk M Dairy ingredients constitute by far the largest volume of ingredients brought into a frozen dessert plant. To help assure consistent quality and safety in dairy ingredients, the farms, transfer stations, receiving stations, and milk plants that supply dairy ingredients need to be routinely inspected and found acceptable by an appropriate regulatory agency. That agency should also verify that the dairy products shipped to this frozen dessert plant were produced, packed, held, and shipped under safeguards equivalent to these guidelines.

All ingredients should be purchased only from suppliers willing to certify or guarantee that their product has been produced and handled in a manner that will assure a safe and wholesome ingredient that will not adulterate the finished product. Some evaluations useful for determining the quality and safety of these ingredients are suggested in the next section of these guidelines.

#### **Specifications**

- Risk M The safety and quality of raw materials can suffer in transit, so a physical check should be made of all incoming ingredients. This check should include an evaluation for conditions related to
  - 1. Product ID and labeling
  - 2. Package condition and integrity
  - 3. Bulging
  - 4. Leaking
  - 5. Dirt, grime
  - 6. Insect infestation
  - 7. Rodent damage
  - 8. Off-odors and nonfood residues in truck or railroad car

Depending on the product received and the problems found, this list can be expanded.

Risk M Improper storage in the plant after receipt can result in contamination. In-plant storage should be monitored for all of the above conditions. Particular attention should be given to resealing containers that have been opened and partially used.

Standards for received dairy ingredients are particularly important to the safety of finished products. Tankers should not be unloaded until the antibiotic, Direct Microscopic Somatic Cell Count (DMSCC), milkfat, cryoscope, titratable acidity, odor, temperature, and appearance checks have been made. A properly staffed lab can perform these checks in 20 to 30 minutes. The potential of losing a silo of milk to antibiotics or an entire run of product to contaminated raw ingredients justifies the time used for proper inspections.

Heating of milk in order to separate it into various cream and reduced fat products is a common practice. It can cause a compromise of product safety if certain precautions are not observed. Raw low-fat milk, skim, or cream that was heated above 45°F but below 160°F for separation may be safely used in frozen desserts if

- 1. It was heated only once prior to pasteurization.
- 2. After separation it was immediately cooled to below 45°F.
- 3. No more than 3 days have elapsed between separation and shipment to the frozen dessert plant.
- 4. If it is heated above 125°F, it meets 30,000 SPC and 10 coliform at plant of shipment, 100 coliform at plant of receipt.

# B. Recommended Dairy Ingredient Specifications

Risk H	Antibiotics	Negative*
Risk H	Other drug residues	Negative*
Risk H	Pesticides, herbicide residues	Negative*
Risk M-H	Titratable Acidity (raw whole,	Not over 0.18%
	lowfat, and skim milk)	
Risk M-H	Temperature	Below 45°F (preferably below 40°F)
Risk M	Appearance and odor	Normal
Risk M	Somatic cells (whole raw milk)	Not to exceed 1,000,000/mL
Risk M	Raw product standard plate count	Not over 500,000/mL
Risk M	Pasteurized standard plate count	Not over 30,000/mL
	(not applicable to cultured products)	
Risk M	Coliform (pasteurized products)	Not over 10/mL except that bulk
	(Also applicable to heat-treated	milk shipments for repasteurization
	raw cream heated to between 125°F	are not over 100/mL at the receiving
	and 161°F)	plant.
Risk M	Phosphatase (pasteurized products)	Less than 1 microgram per mL by
		the Scharer Rapid Method or
		equivalent
Risk L	Added water (not applicable to	None
	reconstituted or recombined	
	product).	

<sup>\*</sup> Negative should be interpreted to include any result below any federally accepted action levels.

# C. Non-Dairy Ingredient Specifications

The specifications for other ingredients are also important to producing safe, high quality frozen desserts and mixes. Some of these items are added after pasteurization. Particular attention should be given to assuring that these ingredients have a sufficiently low water activity, a high enough alcohol content, or are roasted, cooked, heat-treated, or in some way prepared to minimize bacterial growth.

No standards are presented, but action levels consistent with federal guidelines and good industry practices should be set. Product in serious violation of those standards should be rejected.

Some suggested tests for these other ingredients are listed below:

# Chips and Nuts (Cookiebits, etc.)

Risk H	Coliform
Risk H	Chemical/Pesticide Residues
Risk H	Salmonella
Risk H	Aflatoxin
Risk M-H	Visual Inspection
Risk M-H	Foreign Material
Risk M-H	Total Plate Count
Risk M-H	Yeast and Mold
Risk L	Defects and Size
Risk L	Flavor
Risk L	Odor
Risk L	Color

#### Powder and Flavors

Risk H Coliform (if added after pasteurization) Risk H Salmonella (if added after pasteurization) Risk M Total Plate Count Risk M Yeast and Mold Risk L Consistency Risk L **Total Solids** Risk L Brix Risk L Acidity Risk L рН Visual Inspection Risk L Risk L Flavor Risk L Odor Risk L Color

#### Fruits

Risk H	Coliform
Risk H	Pesticide/Chemical Residues
Risk M	Extraneous Material
Risk M	pН
Risk M	Standard Plate Count

Risk M Yeast and Mold Risk L Visual Inspection Risk L Flavor Risk L Color Risk L Odor Risk L Brix Risk L **Defects** Emulsifiers/Stabilizers

- Risk M Extraneous Material
- Risk L Color
- Risk L Flavor
- Risk L Odor
- Risk L Standard Plate Count

#### Colors

- Risk H Coliform (if added after pasteurization)
- Risk H FD&C Approval
- Standard Plate Count (if added after pasteurization) Risk H
- Risk M Extraneous Material
- Risk L Color/Shading
- Risk L Consistency

# Liquid Sucrose/Corn Syrup

- Risk L Color
- Risk L Temperature
- Risk L Visual Inspection
- Risk L Flavor
- Risk L Brix
- Risk L рН
- Risk L Standard Plate Count
- Risk L Yeast and Mold

#### Citric Acid

- Risk L Visual Inspection
- Risk L Color

#### Salt

- Risk L Proper Identity
- Risk L Visual Inspection
- Risk L Flavor/Color
- Risk L Odor

#### Liquid, Dry, or Frozen Egg Yolks

- Risk M **SPC**
- Risk M Coliform

- Risk M Visual Inspection
- Risk L Total Solids
- Risk L Bulging (except liquid)

#### Fruit Juices and Concentrates

- Risk H Chemical/Pesticide Residues
- Risk M Acid
- Risk M Acid/Brix Ratio
- Risk L Specific Gravity
- Risk L Cloud (pectin stability)
- Risk L Yeast and Mold
- Risk L Visual Inspection
- Risk L Flavor/Color
- Risk L Odor

# D. Single-Service Packaging

Sources and Specifications

#### Risk M

If single-service packaging is not constructed of safe materials or arrives at the plant contaminated, plant sanitation and pasteurization safeguards can be compromised.

The bacterial and chemical safety of the packaging and components should be verified. One source of guidance is the "Fabrication of Single-Service Containers and Closures for Milk and Milk Products" (available from U.S. FDA Milk Safety Branch, 200 'C' Street, S.W., Washington, D.C. 20204).

Containers manufactured in accordance with the latest edition of this document should be considered acceptable.

#### IV. GENERAL CONSIDERATIONS

#### A. Product Protection

Cross-Connections

#### Risk H

A cross-connection is any direct piping connection between pasteurized and raw product or any direct piping connection between any food products or ingredients and cleaning and sanitizing solutions.

Cross-connections have caused dairy products to become contaminated with cleaning solutions as well as pathogenic bacteria and have been implicated in major illness outbreaks.

In order to prevent or eliminate cross-connections, a physical break to atmosphere at least as large as the piping diameter is needed.

Blueprints should be reviewed on a periodic basis and updated to reflect existing piping arrangements. This can be accomplished only by "walking" the blueprints through the plant and physically insuring the blueprints are accurate. Internal plant controls are needed to prevent any piping changes without prior review by qualified authorities.

Product and Sanitized Equipment Exposure

Risk H—Pasteurized

Risk M—Raw

Insects, dust, condensate, etc., can carry chemical and bacterial contamination into raw or pasteurized product or onto sanitized product-contact surfaces if these are exposed more than is essential for blending, mixing, and packaging.

In order to prevent this,

- 1. All openings into product or onto sanitized product-contact surfaces should be capped closed, or adequately protected.
- 2. All valves in sanitized lines and lines containing product should be capped or otherwise protected.
- 3. Tank lids should not be propped open during filling (fill line-connections need to be made to tank fittings).
- 4. Long lengths of flexible hose or pipe should not be used to replace rigid product piping. These flexible lines often lie on the floor during use and are difficult to clean and inspect. Often a problem with such a line is observed only when it starts to leak. Short flexible hoses where needed for flexibility and receiving and loadout hoses should not be criticized if they are clean and properly constructed.

#### Hazardous Chemicals

#### Risk M

(Unless actual contamination is observed)

To reduce the risk of accidental contamination, toxic chemicals such as cleaning compounds, sanitizing agents, boiler water compounds, and pesticides need to be stored so that they will not contaminate ingredients, packaging, or finished product. In addition to being separated from food, toxic compounds such as lubricants, boiler water compounds, and pesticides should be stored to minimize the possibility of confusion with cleaning compounds or sanitizing agents or of other misuse in ways that could compromise the finished product.

These precautions should include but not be limited to:

- 1. Only chemicals needed for use in the plant are stored in the plant.
- 2. Toxic chemicals are not stored in any area where food products are received, processed, pasteurized, or stored, or where equipment, containers, or utensils are washed, or where single-service containers and closures are stored.
  - 3. Containers of toxic materials are distinctively labeled.

*Note:* This does not preclude storing detergents and sanitizers convenient to where they are used if they are properly segregated.

### Hand Cleaning Tools

Risk M The use of absorbent items such as rags and sponges should be eliminated to reduce potential harborage and spreading of microorganisms in the plant environment. Separate brushes should be used for product and nonproduct surfaces. Brushes should be nonporous, maintained in good repair, cleaned, sanitized, and stored above the floor between uses. Porous equipment such as wooden handled brushes, tools, paddles, sponges, and cloth should not be used in production areas.

# B. Cleaning and Sanitizing

Safe frozen desserts cannot be produced if ingredients or finished products are permitted to come into contact with containers, utensils, and equipment when they have not been effectively cleaned and sanitized.

- Risk H To accomplish this, product-contact surfaces of all multiuse containers, utensils, and equipment should be clean and should be sanitized before each use.
- Risk M All containers, utensils, and equipment should be cleaned and sanitized at least once each day used; storage tanks should be emptied and cleaned at least each 72 hours.
- Risk H Unclean equipment cannot be effectively sanitized. Soil observed on productcontact surfaces not only provides a place for pathogens to grow, it also affords protection from the sanitizing process.
- Risk H In the case of containers, equipment, and utensils used to handle both raw and pasteurized product without an intervening cleaning, the pasteurized product should be handled first and the equipment effectively cleaned and sanitized after raw product has been handled.
- Risk M Effective cleaning consists of washing, rinsing, and sanitizing. If the washing is done by hand, a two-compartment wash vessel large enough to accommodate the largest piece of equipment cleaned should be provided. If manual chemical sanitizing is done prior to reassembling equipment, a third compartment is needed.
- Risk H Piping equipment and containers used to process, conduct, or package aseptically processed frozen dessert mix beyond the final heat-treatment process should be sterilized before any aseptically processed product is packaged.

# C. Recirculated Cleaning Systems

Risk M

The cleaning of many product-contact surfaces can be evaluated by physical inspection. In the case of equipment disassembled for cleaning, and reassembled for sanitizing, such an inspection is convenient and easy to accomplish. In the case of cleaned-in-place lines, equipment, and silo tanks over 10 feet, such as physical inspection is not normally possible. A record of the cleaning process may be the best and most economical way to evaluate product-contact surface cleanliness.

In the case of pipelines and/or equipment design for recirculated cleaning, the following are needed:

- 1. An effective cleaning and sanitizing regimen for each separate cleaning circuit should be developed and followed.
- 2. A temperature-recording device should be installed in the return solution line after the last equipment washed and before the returning solution is heated to record the temperature and time during which the line or equipment was exposed to cleaning and sanitizing.
- 3. Temperature recording charts need to be identified, dated, and retained for at least one year.
- 4. The recording thermometer for mechanical cleaning systems should be moisture-proof, easy to read and adjust, accurate to within 2°F above 100°F, and protected against

damage to 212°F. The probe should fit inside the pipe without exposed threads. The chart should not move less than one inch per hour.

Provided: A recording device that has been reviewed by the FDA and found to provide sufficient information to evaluate adequately the cleaning and sanitizing regimen and that is approved by the local regulatory agency is an acceptable alternative.

#### Storage Tank Cleaning

Pathogens such as *Listeria* and *Yersinia* can grow in product storage at refrigerated temperatures. For this reason, there is a need to verify that dairy products that will sustain bacterial growth are not held in storage tanks longer than 72 hours prior to pasteurization. Pasteurized mix should be frozen, dried, packaged, or shipped within 72 hours of being pasteurized. If plant production records show tank filling and emptying times, these records may be sufficient, provided that they are kept available for at least as long as the shelf life of the product.

If this is not the case, a 7-day temperature recorder may be needed to measure tank temperature changes. If a recorder is used, the chart should clearly show the times when product was in the tank.

The charts need to be dated and signed or initialed by the operator.

#### Received Bulk Tank Trucks

Risk M Because bulk tank trucks delivering dairy products cannot be inspected for cleanliness, a record is needed of when and where they were last washed and sanitized as well as who did the work. This can be provided on a wash tag from the shipper. If the truck was previously washed at the plant now receiving the product, then the wash chart or cleaning record already on hand is adequate. If bulk tank trucks are washed by hand, a log or other daily cleaning record may be needed.

# D. Construction and Repair of Lines, Containers, and Equipment

When equipment is not constructed so that it can be cleaned easily, or when it is not kept in good repair, it is less likely to be bacteriologically clean. Equipment with unprotected openings can compromise product. Crevices in storage tanks, leaking valves, agitator shafts, shielding, and venting are all areas where pathogenic organisms have been found.

- Risk L-M
- 1. Pitted dairy metal, lead solder, flaking metal-plated, product-contact surfaces that are corroded or rusty cannot be properly cleaned and sanitized and can cause contamination. To prevent this type of problem, multiuse containers and equipment with which milk, milk product, frozen dessert, and frozen dessert mix come into contact should be made of smooth, impervious, corrosion-resistant, and nontoxic material.
- 2. Product surfaces of multiuse containers and equipment may consist of
  - a. Stainless steel of the AISI (American Iron and Steel Institute) 300 series

- b. Equally corrosion-resistant metal that is nontoxic and nonabsorbent
- c. Heat resistant glass
- d. Plastic or rubber and rubberlike materials that are relatively inert and resistant to scratching, scoring, or decomposition. These materials also should be nontoxic, fat-resistant, and relatively nonabsorbent. They should not impart flavor nor odor to the product. They should maintain their original properties under repeated use.
- 3. All joints in containers, equipment, and utensils should be flush and finished as smooth as adjoining surfaces. Where a rotating shaft is inserted through a surface with which milk, milk products, frozen desserts, or frozen dessert mix come into contact, the joint between the moving and the stationary surfaces should be close-fitting. Where a thermometer or temperature-sensing element is inserted through a surface with which milk, milk products, frozen desserts, or frozen dessert mix come into contact, a pressure-tight seal should be provided ahead of all threads and crevices.
- 4. All openings in covers of tanks, vats, separators, etc. are protected by raised edges or other means to prevent the entrance of surface drainage. Condensation-diverting aprons are provided as close to the tank or vat as possible on all pipes, thermometers, or temperature sensing elements, and other equipment extending into a tank, bowl, vat, or distributor, unless a watertight joint is provided.
- 5. If storage-tank agitator motors are located outside of the processing area, the agitator shaft seal should be of a sanitary type that will adequately protect product in the tank.
- 6. All tanks, vats, or vessels used to store product should be equipped with properly constructed indicating thermometers that are accurate in the appropriate temperature range.
- 7. Internal moving parts of freezers and other similar equipment should be constructed so as to keep grease and other contamination from coming into contact with product. They should be constructed so that they are free of cracks and crevices and easy to clean. They should also be demountable for inspection and manual cleaning when necessary.
- 8. All surfaces with which milk, milk products, frozen desserts, or frozen dessert mixes come into contact should be easily accessible or demountable for manual cleaning or designed for mechanical cleaning. All product-contact surfaces should be readily accessible for inspection and be self-draining. Wing nuts, bayonet locks, and similar devices should be used whenever possible in lieu of bolts and nuts to promote easy disassembly.
- 9. There should be no threads used in contact with milk, milk products, frozen desserts, or frozen dessert mixes except where needed for functional and safety reasons, such as in clarifiers, pumps, and separators. Such threads should be of a sanitary type.
- 10. All multiuse containers and other equipment should have rounded

- corners, be in good repair, and be free from breaks, crevices, and corrosion. Milk cans should have umbrella-type covers.
- 11. Except where required for essential functional reasons, strainers, if used, should be of perforated metal design and so constructed as to utilize single-service strainer media. Multiple-use woven material should not be used for straining.
- 12. All product vessels, such as tanks, vats, blending horns, and reclaim dump vats, should be provided with properly constructed covers for all openings. These should be in place whenever frozen desserts or components of frozen desserts are inside. A cover may properly be removed for inspection or hand addition of products. Fill lines should not block nor prop these covers open.
- 13. Sanitary piping, fittings, and connections should be designed to permit easy cleaning, kept in good repair, and free of breaks or corrosion, and contain no dead ends of piping in which product may collect.
- 14. All interior surfaces of demountable piping, including valves, fittings, and connections, should be designed, constructed, and installed to permit inspection and drainage.
- 15. Pasteurized product should be conducted from one piece of equipment to another only through sanitary milk piping.
- 16. All cleaned-in-place milk pipelines and return solution lines should be rigid, self-draining, and supported to maintain uniform slope and alignment. Flexible hoses of proper construction may be used to receive products from a bulk tank truck; also, short flexible hoses may be needed to bring product to weigh tanks or portable equipment. Return solution lines should be constructed of material meeting the specifications of item 2 above. If gaskets are used, they should be self-positioning, of material meeting the specifications outlined in item 2 above, and designed, finished, and applied to form a smooth, flush interior surface. All interior surfaces of welded joints in pipelines should be smooth and free from pits, cracks, and inclusions.

Welds in welded lines should be inspected and approved by the regulatory agency.

Each cleaning circuit should have access points for inspection in addition to the entrances and exits. These may be valves, removable sections, fittings, or other means of combination that are adequate for inspection of the interior of the line. These access points should be located at sufficient intervals to determine the general condition of the interior of the line.

Detailed plans for welded pipeline systems should be submitted to the regulatory agency for written approval prior to installation. No alteration or addition should be made to any welded milk pipeline system without prior written approval from the regulatory agency.

Risk H 17. Strong odor and product buildup beneath the underside of a tank can indicate tank leakage. Such a tank should be inspected to find the problem. If the problem is a leak in the metal lining of the tank, the tank should be taken out of service until it is repaired.

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# E. Separation of Operations

Risk M

Some dairy plant operations are incompatible and should be separated. Pathogens have been found in finished products from plants where packaging materials were transferred, unprotected, through garbage and case-wash areas.

Packaging and ingredients have been water-damaged to the point of being unusable when stored in processing areas.

The general rule is that if product or packaging in one operation can be put at increased risk by having another operation near, then an adequate separation is needed.

The following areas should be separate:

- 1. The tank truck receiving area
- 2. The processing area
- 3. The can- or case-wash areas
- 4. The dry storage area
- 5. The packaging area
- 6. Other areas as needed

# F. Dry Storage

- Risk H Damaged packages need to be evaluated to determine if the product inside is contaminated. Contaminated ingredients and packaging should be discarded.
- Risk M Ingredients and packaging should be stored off the floor and physically separated from cleaners and similar toxic compounds. Cleaning compounds need to be separated from other toxic chemicals.
- Risk M Dry storage areas should be kept reasonably clean and free of clutter. Materials should be stored away from walls far enough to allow adequate cleaning.

Dry storage areas should be provided with enough light to see developing housekeeping or pest problems.

Open or partially used packages of ingredients or containers should be resealed or otherwise adequately protected.

#### G. Cooling

- Risk M–H When frozen dessert mix and some components of frozen dessert mix are not maintained at below 45°F the bacterial content, including pathogens, can increase. Therefore, all milk, milk products, frozen dessert mix, liquid eggs, and dairy ingredients should be maintained at 45°F or below. After such products are raised above 45°F for blending, separation, or pasteurization, they should be immediately cooled to below 45°F.
- Risk M All storage tanks and refrigerated storage rooms should be equipped with accurate thermometers so that this can be verified.
- Risk M Some plate or tubular coolers use pasteurized products to cool or heat raw products in the same manner as the regenerator section of an HTST pasteurization system. In this case, safeguards similar to those in an HTST regenerator need to be provided to prevent pasteurized product contamination in the event of a leak.

# H. Refrigerated Storage Rooms

Mix should be protected from contamination and temperature rise during loading. Trucks used to transport packaged mix should be clean and tight and maintain the mix below 45°F.

- Risk H Product in coolers should be stored below 45°F.
- Risk M In order to be able to monitor coolers properly, accurate thermometers need to be placed in the warmest areas of refrigerated storage.
- Risk M–H Pathogens have been found in drainage and condensate from cooling units and in condensate from ceilings; therefore this type of dripping should not be allowed to fall onto packaged mix or ingredients in the cooler. Cooler walls and ceilings need to be kept in good repair, clean, and free of mold.
- Risk M Cooler floors and drains need to be kept clean and in good repair. Pathogens such as *Listeria* sp. have been found in cooler drains, pooled water on cooler floors, and under loose metal floor plates. Pathogens have also been found in tracks and track pits that have not been kept clean. Keeping the floor in these areas in sound condition and clean is also needed.
- Risk M Track lubrication should contain an effective sanitizer wherever practical.

# I. Building Maintenance and Construction

Risk L

(Unless it is related to an existing sanitation problem)

Building and grounds construction and maintenance should facilitate sanitary operation and can go a long way toward easing or preventing problems with environmental contamination and aid in pest control.

The following areas should be considered:

- 1. Painted surfaces should be kept in good repair. Lifted or peeling paint can collect moisture and harbor insects.
- 2. Beams and piping in the processing or storage room should be clean and free of rust.
- 3. Insulation should not be worn nor torn. Poorly maintained insulation can harbor pests or get into ingredients or finished products.
- 4. Ledges and other horizontal surfaces should be kept clean.
- 5. Windows should be in good repair to prevent insect or rodent access.
- 6. Screens should be in place and in good repair.

#### J. Grounds and Roof

The grounds surrounding a frozen dessert plant should be kept in a condition that will protect against product contamination.

Risk L

(Unless it is related to an existing problem inside the plant)

1. The building exterior is the first line of defense against pests. The exterior needs to be free from tall weeds, trash, and discarded equipment. Sparse landscaping

- should be stressed. Low-lying areas that collect stagnant water should be eliminated.
- 2. Auxiliary buildings, such as storage sheds, should not be havens for pests. The buildings should be properly maintained.
- 3. Outside drains and sewer lines should not back up. All drain covers need to be in place and in good repair.
- 4. The roof should be in good repair without pooled water. Drains, gutters, and drainpipes should be clean. The roof should not be cluttered or full of debris. Pooling water will eventually cause a roof to sink, and then stagnant, contaminated water can leak into the plant.
- 5. Product spillage should be minimized; areas where spilling occurs should be kept clean. Particular attention should be given to the trash-compactor area, the truck-loading area, and the returned product handling area.
- 6. Trash containers should be covered and should be emptied at regular intervals.
- 7. Any exterior openings that are not essential should be sealed. Air filters, screens, or air curtains should be provided as required.
- 8. Provision should be made for washing of truck and trailer interiors with drainage to a sanitary sewer.
- 9. Vehicles should be parked on paved areas only.
- 10. Bird roosting areas should be eliminated.

# K. Plant Environment

The general plant environment should be recognized as having a significant impact on the safety of finished product. Special consideration of refrigerated areas is necessary. Organisms such as *Listeria* and *Yersinia* grow at refrigerated temperatures. Aerosols may act as vehicles in which organisms such as *Listeria* and *Yersinia* may contaminate exposed product and product-contact surfaces. *Listeria* has been frequently isolated from floor drains in processing and other areas.

- Risk M Keeping floors, walls, and ceilings clean, relatively dry, and free from condensate buildup is imperative in order to minimize product contamination. Special attention should be given to the cleaning and sanitizing of conveyor track and belt systems. Cleaning should not take place during production runs when product and/or product-contact surfaces are exposed.
- Risk M The pooling of milk, water or other processing wastes should be minimized. Areas such as ducts, cracks, holes, spaces under loose metal floor plating, etc. should be given special attention.
- Risk M Returned goods should be isolated in a properly identified holding area.
- Risk M Practices that may lead to the formulation of aerosols such as the use of high pressure hoses and unshielded pumps should be minimized or eliminated.
- Risk M Floor drains should not be located under or in close proximity to filling and packaging equipment. Floors and drains should be constructed and maintained to insure proper drainage. Brushes used for cleaning floor drains should not be used for any other purpose and should be cleaned and stored in proper strength sanitizing solution between uses. Floor drains should be frequently cleaned and periodically flushed with a sanitizing solution. Floor drain covers and baskets should be cleaned and sanitized after each production run.

Risk H Under *no* circumstances should high-pressure hoses be used to clean drains.

#### L. Pest Control

- Risk H Human pathogens including *Listeria* have been found on flies, roaches, rodents, and even ants. Insects and rodents can carry these pathogens to sanitized surfaces and clean areas. Control of these pests is a vital part of any plant program to combat pathogen infections.
- Risk H Plant areas should be essentially free of insects, rodents, and other pests.
- Risk M Prevention remains the best control measure. Implementing the buildings and grounds sections of these guidelines will go a long way toward that protection. In most plants, however, prevention should be supplemented with an effective treatment program. Pesticides should be applied by a trained individual and in conformance with applicable state and federal law. Each plant should have full material safety data sheets and EPA registration for all pesticides used in the plant.
- Risk H In order to reduce the possibility of accidental contamination, these products should be stored separately from food ingredients and cleaning compounds. The materials should be stored under lock and key, and the storage locker should be clearly marked "Warning Pesticide Storage." A log of pesticide usage and a diagram showing where residual pesticides are routinely applied should be accurately maintained.
- Risk M The use of rodenticide within the plant should be discouraged to minimize the chances of product contamination. Rodent control should be preventive with traps placed both inside and outside the plant.

#### M. Toilet and Sewage Disposal

Toilet rooms and improper sewage disposal methods can be sources of potentially serious contamination.

- Risk H Sewage and other liquid waste should be disposed of in a sanitary manner.
- Risk M Toilet rooms should be completely enclosed and have tight-fitting, self-closing doors. Dressing rooms and toilet rooms should be kept clean, well lighted, and adequately ventilated.
- Risk M If contamination is to be minimized, toilet rooms should not open directly into rooms where product is exposed or packaged.

#### N. Hand Washing Facilities

Risk M Proper use of hand washing facilities is essential to personal cleanliness and reduces the likelihood of contamination of frozen desserts.

To be effective, hand washing facilities should

- 1. Have hot and cold (or warm) running water, soap, and individual sanitary towels or other effective hand drying devices.
- 2. Be convenient to all toilets and to all rooms in which milk plant operations are conducted.
- 3. Be kept in a clean condition and in good repair.

# O. Water Supply

Risk M–H The importance of an adequate supply of safe water is difficult to overestimate. The effectiveness of plant cleaning is dependent upon an adequate supply of safe water. Contaminated water could contaminate product.

The water supply should be accessible in order to encourage its use in cleaning operations; it should be adequate so the cleaning and rinsing is thorough; and it should be of safe, sanitary quality in order to avoid the contamination of product or milk equipment and containers.

A private water supply is considered adequate when it is properly located, protected, operated, and of a safe sanitary quality. Parts of the systems, such as well seals and storage tanks, should be easily accessible for inspection.

A municipal supply is considered to be safe as it enters the plant if it has been evaluated and is approved by the state water control authority.

Water at a plant faucet even from an acceptable municipal source cannot be considered as safe if there are any cross-connections in the plant between safe water and unsafe or questionable water. An unprotected connection between a safe water system and such things as product valve clusters, sweetwater lines, cooling tower lines, and boiler chemical feed lines constitutes a potential cross-connection. Cross-connections may also exist when an unprotected safe water inlet pipe is below an effective overflow on cooling tower reserve tanks, boiler water treatment chemical reserve tanks, CIP chemical makeup tanks, sweetwater or glycol tanks, or any other container.

Safe water systems can be effectively protected from such cross-connections if an effective backflow preventer acceptable to the state water authority is properly installed and operating between the safe water system and an unsafe or questionable source.

*Note:* Water from municipal or plant systems that violate national, state, or local plumbing codes is not acceptable until the violations are corrected.

If the water used by a plant is from private wells or springs, these should be individually evaluated. Excellent guidance for this evaluation can be found in the EPA publication, *Manual of Individual Water Supply Systems*, EPA-430/9-74-007. Guidance already tailored to dairy industry needs can be found in Appendix D (standards for water sources) and Appendix G (chemical and bacteriological tests) in the latest edition of the *Grade A Pasteurized Milk Ordinance* published by the U.S. Food and Drug Administration.

Water Reclaimed from the Condensing of Milk and Milk Products

- Risk M Reclaimed water may be used to wash equipment as well as other similar uses, but the potential exists for it to be contaminated. If this water is to be safely used, the following criteria should be applied.
  - 1. The reclaimed water should be free of coliform and not exceed a total plate count of 500 per milliliter.
  - 2. Coliform and bacteria samples should be collected daily for two weeks following initial approval of the installation and at least semiannually thereafter, provided that daily tests should be conducted for one week following any repairs or alteration to the system.
  - 3. The organic content should be less than  $12 \,\mathrm{mg/L}$  as measured by the chemical oxygen demand or permangate-consumed test; or have a standard turbidity of less than 5 units.
  - 4. Automatic fail-safe monitoring devices should be used to monitor and automatically divert (to the sewer) any water that exceeds the standard.
  - 5. The reclaimed water should be of satisfactory organoleptic quality and have no off-flavors, odors, or slime formations.
  - 6. The reclaimed water should be sampled and tested organoleptically at weekly intervals.
  - 7. Approved chemicals, such as chlorine, with a suitable detention period, may be used to suppress the development of bacterial growth and prevent the development of tastes and odors.
  - 8. The addition of chemicals should be by an automatic proportioning device prior to the water entering the storage tank to assure satisfactory quality water in the storage tank at all times.
  - 9. When chemicals are added, a daily testing program for such added chemicals should be in effect. Added chemicals should not add substances that will contribute to product contamination.
  - 10. The storage vessel should be properly constructed of such material that it will not contaminate the water and can be satisfactorily cleaned.
  - 11. The distribution system within a plant for such reclaimed water should be a separate system with no cross-connections to a municipal or private water system.
  - 12. All physical, chemical, and microbiological tests should be conducted in accordance with the latest edition of *Standard Methods for the Examination of Water and Wastewater*.

#### P. Personnel

Improper employee practices and traffic patterns have resulted in pathogen contamination of finished product.

Employee Practices

Risk H Employees should be instructed to wash their hands after using the toilet. Toilet rooms should be located to minimize cross traffic (processing room employees crossing raw receiving or case wash areas and vice versa).

Clothing or other personal belongings should not be stored in processing areas. Caps, hats, hair nets, beard nets should be worn in such a manner that they are effective hair restraints.

Employees who work with food products, packaging, or food-contact equipment need to be adequately trained in hygienic practice for their particular duties. Each plant should have personnel who are responsible for identifying sanitation failures. These people should have sufficient experience and/or formal training to provide the level of competency needed for the production of clean, safe food. Employees with obvious illnesses, infected cuts, or abrasions, etc. should be excluded from working in processing areas or performing other functions can contaminate product, product-contact surfaces, or packaging material.

Risk M The use of tobacco products, chewing gum, or other food for employee consumption should not be permitted in any production area. Employees should not be allowed to wear hairpins, rings, watches, etc. in production areas. Special attention is needed to assure that street clothes are not allowed in the processing area and that plant clothing (including rubber boots) do not leave the plant. It is recommended that the laundering of all work clothing should be the plant's responsibility and proper procedures for storing and issuing clean clothing need to be developed. Of equal concern is a potential problem associated with plant maintenance personnel and others working in raw milk areas and then working on/near pasteurized milk equipment without adequate cleanup of hands, tools, clothing, etc.

It is recommended that uniforms be color-coded by department to control movement of employees into restricted areas. When disposable single-service gloves are necessary to handle exposed product-contact surfaces during a production run, they should be maintained in clean and sanitary condition. Single-service gloves should be thrown away whenever they become torn, contaminated, or removed for any reason.

Hand washing facilities should be properly designed and conveniently located near work stations. Employees should be encouraged to use them frequently.

# Plant Traffic

Risk M Employees should be trained to recognize the importance of cross-contamination problems within the plant. Special emphasis in training employees is needed to avoid the spread of pathogens within the plant environment from outside the plant (home/farm, etc.) or from areas such as the machine shop, raw milk receiving area (manure from farms can be carried on trucks of raw milk). Employees should understand that organisms can be carried on their clothing, boots, tools, etc. A traffic pattern of restricting access to processing areas should be in place. Milk haulers and all other nonprocessing operations people should be restricted from entering the processing areas. Foot baths should be routinely monitored for proper disinfectant strength and cleanliness. A continuing review and restriction of the movement of pallets, forklifts, and other similar equipment from raw milk, case wash, dock or other such areas into processing/packaging areas is needed. Wooden pallets have been found contaminated with organisms such as *Listeria* and *Yersinia*.

#### V. PLANT SYSTEMS

#### A. Air Under Pressure—Product Contact

Risk M Air, when used for agitation, air blows, and incorporation into product (overrun) is strongly suspected as a vehicle for allowing pathogenic organisms to enter product.

Improperly protected air can also lead to product contaminated with particulate matter, condensate, or oil.

Processing systems that incorporate air directly into the product, such as freezers, air blows, and air agitation systems, should be designed to reduce potential contamination and should be easily cleanable. A process air system should contain appropriate filters to remove undesirable particulate matter. Sanitary check valves should be provided as necessary to prevent product backup into air lines. Air blow and agitation equipment should be routinely checked for proper assembly and cleanliness. Most sanitary check valves, air blows, and agitation equipment are not satisfactorily cleaned by the usual CIP methods and should be dismantled and manually cleaned and sanitized routinely.

A summary of technical requirements for safe, dry sanitary air listed in "3-A Accepted Practice for Supplying Air Under Pressure in Contact with Milk, Milk Products and Product-Contact Surfaces" is presented below.

Air Supply Equipment—The compressing equipment is designed to preclude contamination of the air with lubricant vapors and fumes. Oil-free air may be produced by one of the following methods or their equivalent:

- 1. Carbon ring piston compressor
- 2. Oil-lubricated compressor with effective provision for removal of any oil vapor by cooling the compressed air
- 3. Water-lubricated or nonlubricated blowers

The air supply should be taken from a clean space or from relatively clean outer air and passed through a filter upstream from the compressing equipment. This filter is located and constructed so that it is easily accessible for examination, and the filter media are easily removable for cleaning or replacing. The filter should be protected from weather, drainage, water product spillage, and physical damage.

Moisture Removal Equipment—If it is necessary to cool the compressed air, an after-cooler should be installed between the compressor and the air-storage tank for the purpose of removing moisture from the compressed air.

Filters and Moisture Traps—Filters are constructed so as to assure effective passage of air through the filter media only.

The air under pressure passes through an oil-free filter and moisture trap for removal of solids and liquids. The filter and trap are located in the air pipeline downstream from the compressing equipment and from the air tank, if one is used. Air pipeline filters and moisture traps downstream from compressing equipment are not needed where the compressing equipment is of the fan or blower type.

A disposable media filter is located in sanitary air pipelines upstream from and as close as possible to each point of application or ultimate use of the air.

Air Piping—The air piping from the compressing equipment to the filter and moisture trap is readily drainable.

A product-check valve of sanitary design is installed in the air piping downstream from the disposable media filter to prevent backflow of product into the air pipeline, except that a check valve should not be required if the air piping enters the product zone from a point higher than the product overflow level that is open to the atmosphere.

Air distributing piping, fittings, and gaskets between the terminal filter and/or product-contact surface need to be of sanitary milk piping. When the air is used for such operations as removing containers from mandrels or moving other types of packaging, other nontoxic piping may be used.

Filter Media—Air intake and pipeline filters should consist of fiberglass, cotton flannel, wool flannel, spun metal, electrostatic material, or other equally acceptable filtering media, which are nonshedding and which do not release to the air toxic volatiles, or volatiles that may impart any flavor or odor to the product.

Disposable media filters consist of cotton flannel, wool flannel, spun metal, nonwoven fabric, U.S.P. absorbent cotton fiber or suitable inorganic materials, which under conditions of use are nontoxic and nonshedding. Chemical bonding material contained in the media is nontoxic, nonvolatile and insoluble under all conditions of use. Disposable media should not be cleaned and reused.

Filter Performance—The efficiency of intake filters should be at least 50% as measured by the National Bureau of Standards "Dust Spot Method" using atmospheric dust as the test aerosol. The efficiency of either air pipeline filter or disposable filters should be at least 50% as measured by the DOP (dioctyl 1-phthalate fog) test.

The above does not apply when the compressing equipment is of the fan or blower type.

#### B. Steam Standards

Risk M Stream is used to provide heat for vat and HTST pasteurization processes. Vat linings and HTST plates can leak if defective. The defects are very hard to determine by physical inspection. Cleaning solutions and sanitizers are heated by steam. Steam may also be used directly against product in some applications. If the steam is not safe and the steam system not provided with certain minimum safeguards, the result can be chemically contaminated finished products.

The minimum requirements for safe steam are

Source of Boiler Feed Water

Potable water or water supplies acceptable to the regulatory agency should be used.

Feed Water Treatment

Feed waters may be treated, if necessary, for proper boiler care and operation. Boiler feed water treatment and control should be under the supervision of trained personnel or a firm specializing in industrial water conditioning. Such personnel should be informed that the steam is to be used for culinary purposes. Pretreatment of feed waters for boilers or steam-generating systems to reduce water hardness before entering the boiler or steam generator by ion exchange or other acceptable procedures is preferable to addition of conditioning compounds to boiler waters. Only compounds complying with Section 173.310 of Title 21 of the Code of Federal Regulations may be used to prevent corrosion and scale in boilers or to facilitate sludge removal.

Greater amounts should not be used of the boiler water treatment compounds than the minimum necessary for controlling boiler scale or other boiler water treatment purposes. No greater amount of steam should be used for the treatment and/or pasteurization of frozen dessert mix, milk, and milk products than necessary.

It should be noted that tannin, which is also frequently added to boiler water to facilitate sludge removal during boiler blow down, has been reported to give risk to odor problems and should be used with caution.

Boiler compounds containing cyclohexlamine, morpholine, octadecylamine, diethylaminoethanol, trisodium nitrilotriacetate, and hydrazine should not be permitted for use in steam in contact with frozen dessert mix.

# Boiler Operation and Piping Assemblies

A supply of clean, dry saturated steam is necessary for proper equipment operation; boilers and steam-generation equipment should be operated so as to prevent foaming, priming, carryover, and excessive entrainment of boiler water into the steam. Carryover of boiler water additives can result in the production of mix with off-flavors. Manufacturer instructions regarding recommended water level and blowdown should be consulted and rigorously followed. The blowdown of the boiler should be carefully watched so that an overconcentration of the boiler water solid and foaming is avoided. It is recommended that periodic analyses be made of condensate samples. Such samples should be taken from the line between the final steam-separating equipment and the point of the introduction of steam into the product.

See Figures 29 and 30 of Appendix H in the current edition of the Grade A Pasteurized Milk Ordinance for Suggested Steam Piping for Air Space Heating and Defoaming as well as for steam piping for steam injection and infusion.

Boilers used to produce steam for injection into HHST pasteurizer holding tubes are equipped with a deaerator to remove noncondensable gases.

#### C. Recirculated Cooling Water and Glycol

Risk M Recirculated cooling water (sweet water) and recirculated glycol and water mixtures are often used to cool frozen dessert mix or dairy ingredients. No practical method now exists to assure that pressure exerted by the cooling water or glycol in these coolers will always be less than the pressure of the product being cooled. Contamination of product has been caused by *Listeria*-contaminated sweet water as a result of leaking plates.

Glycol solutions can also support pathogen growth. Storage tanks, jacketed vessels, cooling plates, etc. occasionally leak. Therefore a thorough check should be made of sweet water and glycol cooling systems to assure that reserve tanks are protected against the entrance of contamination and that the cooling media are not exposed anywhere in the system.

The water or glycol and water mix from each of these cooling systems should be sampled and tested at least each 6 months and found to be free of coliform. This water or glycol should also be tested for pathogens such as *Listeria* sp.

# D. Heating, Ventilation, and Air-conditioning HVAC Systems

Risk M–H Airborne contamination is strongly suspected as a vehicle for allowing pathogenic organisms to enter product. A comprehensive assessment of both processing and ventilating air utilized within the plant should be conducted. Heating, ventilating, and air-conditioning (HVAC) systems should be designed for easy cleaning and should be periodically cleaned. Condensate drip pans and drain lines should be periodically checked and cleaned to assure they are not providing favorable environments for the growth of pathogenic organisms. Air systems in refrigerated areas should also be designed for ease of cleaning and should be routinely cleaned. All plant areas should be kept reasonably dry and free of mold, algae, and odors.

HVAC systems should be properly designed and adjusted to maintain positive pressure in areas where product is exposed, such as batching, freezing, filling, and packaging operations. Air transfer between potentially contaminated areas such as raw-product blending or ingredient storage and packaging areas should be minimized.

Risk M Outside air should be filtered and free of condensate. Airflow should be determined and controlled to eliminate direct air movement blowing onto product or product-contact surfaces or filling and packaging areas. Air filters should be of the type effective in removing particulate matter and condensate, thus reducing the potential for dispersion of microorganisms. Filters should be kept clean and replaced according to an established maintenance schedule.

Processing systems that incorporate air directly into the product, such as freezers, air blows, and air agitation systems, should be designed to reduce potential contamination and should be easily cleanable. Process air systems should meet the criteria in these guidelines.

Appendix B provides details on special operations.

# 36

# **Frozen Foods and Enforcement Activities**

# **Peggy Stanfield**

Dietetic Resources, Twin Falls, Idaho, U.S.A.

# I. FRESH AND FROZEN ORANGE/OTHER JUICE: ESTABLISHMENT INSPECTION

The sanitation inspection of orange juice plants over the years has evolved to include an investigation for possible intentional adulteration.

#### A. Juice Adulteration

Criminal investigations of juice adulterators have shown that as analytical capabilities have improved adulteration methods have become more sophisticated. While U.S. Food and Drug Administration (FDA) criminal cases have focused on orange juice, it is possible for an unethical firm to gain a market advantage over honest competitors, and to defraud consumers, through the intentional adulteration of almost any juice or percentage juice drink. Therefore, when inspecting any fruit juice or percentage juice drink manufacturers, you should be alert for any evidence of economic fraud.

The primary market advantage to juice adulteration is the cost advantage the manufacturer can realize if he can extend the product or replace some or all of the juice ingredient(s) with ingredients of lesser value. There are many different variations on such adulteration including

- 1. Dilution with water. In reconstituted juices, excessive simple dilution can be detected by a simple Brix measurement (% by weight of soluble solids), which measures the percentage of fruit sugars in the product. The addition of water reduces the Brix measurement. However, even the most sophisticated economic adulteration is ultimately designed with the addition of inexpensive water in mind. The extra steps taken by the sophisticated adulterator are designed to conceal the addition of water by making the water appear to be the water inherent in the fruit juice.
- 2. Addition of sugar and water. Sugar is used to mimic the natural fruit sugar and to conceal the addition of water from a Brix measurement. Any sugar ingredient can be used as a substitute for the juice sugars to defeat the Brix test. A manufacturer making or blending juice concentrates may add only the sugar, since the additional water will be added later when the product is reconstituted. Sweeteners from cane and corn are the least

expensive sweeteners and offer the most economic advantage to a manufacturer. However, cane or corn sweeteners can be detected in orange or apple juice by using a carbon stable isotope analysis. Thus the more expensive invert beet sugar has been more commonly used as analytical methods have become more sophisticated. Less sophisticated formulations for sugared juices may only involve sugar substitution for the juice ingredient(s) and the addition of extra water, while more sophisticated formulations will contain other adulterants in order to conceal the addition of the sugar. For instance, adulterated orange juice or grapefruit juice may have added amino acids to make the protein profile appear normal, citric acid to adjust the acid ratio, and/or trace minerals to make the chemical profile appear more normal.

- 3. Adulterated apple juice may have added malic acid and/or trace minerals. Lemon juice may be adulterated with citric acid and may contain added sugar.
- 4. Addition of pulpwash solids and water. Pulpwash is the residue exhaustively extracted by repeated water washing from the previously pressed orange (or grapefruit) pulp used to manufacture the fruit juice. Although it contains
- 5. Orange solids, it is an inferior product to concentrated pressed juice and it is not a kind of orange juice. It is sold as a concentrate
- 6. And is considerably less expensive than orange juice solids. Frozen concentrated orange juice (FCOJ) and products made from FCOJ may contain the in-line pulpwash from the same batch of oranges used to make the juice concentrate. However, addition of pulpwash, under other circumstances is not permitted. Fresh juice and pasteurized juice cannot contain in-line pulpwash, and the pulpwash obtained from their manufacture is sold separately for use in drink manufacture. The State of Florida requires all pulpwash manufactured in Florida to contain the marker sodium benzoate, an ingredient
- 7. Not permitted in orange juice. Thus the presence of sodium benzoate in an orange juice is suggestive of the addition of pulpwash.
- 8. A less expensive juice like apple, pear, pineapple, or white grape, or an inexpensive decharacterized juice (a fruit juice such as deflavored, decolored white grape or pineapple juice), may be substituted in part for a more expensive juice.
- 9. One juice may be made to appear to be another juice which sells for a higher price. For example, an artificial color or another juice such as plum or pomegranate juice may be added to white grapefruit juice to make it appear to be pink/red grapefruit juice. An expensive grape juice (such as concord) might be extended with a cheaper, less desired grape juice (such as white grape) in order to obtain a higher market price.
- 10. A preservative, especially one not approved for use in juices or drinks, may be added to extend the shelf life while saving plant cleanup, repair, and maintenance costs. Orange juice may not contain added preservatives.
- 11. A drink may declare added juice as a percentage of the ingredients, although it may not contain that percentage of juice, may not contain the specified juice, may contain a decharacterized juice, or may not contain any juice ingredient.
- 12. Pasteurized or reconstituted juice may be labeled as fresh squeezed juice. A conspiracy between firms to adulterate juice may be concealed through the use of code names for products or through the production of a drink ingredient at one plant/firm which is ultimately used for the production of a juice at another plant/firm.

Creative investigational techniques may be needed to detect juice adulteration. An investigator familiar with plumbing may be needed to trace the pipe and valve system. The building layout may need to be evaluated for secret tanks/rooms/pipes. Plant surveillance may be necessary to look for unusual delivery/shipping patterns and/or for off-site storage/production facilities. It is not unusual for a plant that is adulterating juices never

to be in production during an inspection. Thus a lack of production during your inspection may indicate an attempt to conceal certain illegal activities.

Under current law, food firms are not required to reveal formulations or show production records. Our criminal investigations have shown that illegal ingredients may well be added to juice even though the formulations given to the FDA during inspections do not reveal their use. Thus inspectional technique becomes critical to the detection of juice adulteration.

Look for the presence of likely adulterants in the plant, i.e., sugar, sugar syrups, invert syrups, pulpwash, decharacterized or other inexpensive juices, malic acid, or other acidulants. These materials may also be stored in tankers or other trucks on or near the premises. Frequently, a plant engaging in economic adulteration will manufacture a line of drink products which uses the likely adulterants so that their presence in the plant can be explained to an FDA investigator.

Probe the uses of any likely adulterants, any unlabeled/unidentified/coded products used, quantities purchased, quantities of explanatory product manufactured, frequency of manufacture, etc.

Be particularly alert to the presence of beet sugars and beet invert syrups as very suspicious. Manufacturers using a sweetener in a separate line of drink products can usually make a drink product of the same quality more cheaply with cane or corn sweeteners. The primary attraction of beet sugar and beet invert syrups is the difficulty of detecting them in finished juice products. Look for the presence of unexplained juice concentrates, i.e., white grape or pear juices, or pulpwash in the plant. Pulpwash is often identified by a name other than pulpwash such as orange solids, water extracted orange solids, WESOS, etc.

#### **B.** Raw Materials

- 1. Prepare a list of all raw materials and their suppliers.
- 2. Make a list of all additives found and their suppliers. It may be helpful to inventory all additives and perform an audit of their use in
- 3. Legal products.
- 4. Determine what types of sugars are in stock and their uses.
- 5. If orange pulpwash solids are in stock, determine their source and how they are
- 6. Try to determine if off-site storage facilities are owned or rented. These sites should be included in the inspection.

#### C. Manufacturing

- 1. If pulp or other additives are used, identify the point at which they are added to the juice. Look for containers in the area that might suggest other additives. Look at incoming products and talk to the haulers.
- 2. Obtain copies of pertinent production records covering products manufactured during the inspection and for all products sampled. These records may contain code names. Study them and try to determine irregularities. Get definitions for all terms. Elicit a statement from an appropriate person as to other ingredients used that are not listed and document the answer. Compare batch production records with the actual manufacturing operation.

- 3. Watch the manufacturing operation to identify possible irregularities. If adulteration is suspected, try to identify the person who is keeping the receiving, shipping, and production records and where those records are stored.
- 4. If illegal use of pulpwash solids or other sugars is suspected, review any available records and attempt to determine whether the amount of juice solids received is consistent with the amount of juice solids produced. Document findings.
  - 5. Collect Official Samples to document the receipt and use of any adulterants.
  - 6. If equipment contains product or slime build up, report and take scrapings.

#### II. FROZEN STRAWBERRIES: ESTABLISHMENT INSPECTION

The FDA has issued the following guidelines during the inspection of an establishment that manufactures frozen strawberries.

Direct special attention to the following areas. Prompt and careful handling of strawberries is essential because of rapid ripening and susceptibility to mold. Since the quality of the fruit deteriorates rapidly after picking, especially in hot weather, improper handling and transporting, or processing delays, may result in the deterioration of good quality raw materials.

#### A. Raw Materials

Check the quality of incoming berries for contamination with sand, mold, or rot, as follows:

- 1. Examine 100 berries selected at random from product in storage and each lot delivered during the inspection. Cut all berries in half and examine for rot. USDA Bulletin 2140, "Strawberry Diseases," is a useful guide to various types of rot. List grit on washed and sorted berries.
- 2. Make sufficient periodic examinations to determine the overall quality of the raw stock being used. If 5% or more berries contain rot, evaluate any processing delays, the amount of static fruit on hand, and the conditions under which it is held.
- 3. Determine growing conditions in the area such as disease, insects, and weather bearing on the availability of fruit.

#### B. Processing

- 1. Collect 100 berry samples from each sorting line. Cut all berries in half and examine for rot. Make sufficient periodic examinations of sorted berries to determine overall quality of berries being packed.
- 2. Determine if unfit fruit (moldy, rotten, etc.) is blended with good fruit in the dumping or packing operations to form an adulterated product.
- 3. Determine the deposition of unfit fruit (trash, animal feed, etc.).
- 4. Evaluate any delays in the processing flow that allow in-process berries to rot or become moldy.
- 5. If processing equipment contains slime or buildup, report and take scrapings for analysis.
- 6. Evaluate the firm's packaging and freezing operation.

#### C. Sample Collection

Collect samples of raw materials and finished products to document

- 1. The presence of sand, grit or other contaminants in the finished product.
- 2. When belt pick-outs, after sorting, run 5% or more definite rot spots.
- 3. Trade or consumer complaints, which may indicate production of a violative pack.
- 4. Factory evidence of the production of strawberry juice from rotten berries.

#### III. FROZEN MEAT AND POULTRY RECALLS

The U.S. Department of Agriculture (USDA) regulates the safety of meat and poultry products, including recalls. To illustrate the types of potential hazards associated with fresh and frozen products, some examples of recalls are provided here.

The USDA implements the following recall classifications:

Class I This is a health hazard situation where there is a reasonable probability that the use of the product will cause serious, adverse health consequences or death.

Class II This is a health hazard situation where there is a remote probability of adverse health consequences from the use of the product.

Class III This is a situation where the use of the product will not cause adverse health consequences.

Examples of recalls are as follows:

Frozen beef products: Class III Recall (November 20, 2002)

Company: Famous Chili Inc., Fort Smith, Arkansas

Date and distribution: Produced between Oct. 1, 2000, and Nov. 19, 2002. Distribution: Hotels, restaurants and institutions in Arkansas and Oklahoma

Product and amount: An undetermined amount of frozen beef products

Establishment Code: "EST. 10647"

Reason: Contains an undeclared ingredient, monosodium glutamate (MSG). MSG is used as a flavor enhancer in a variety of foods prepared at home, in restaurants, and by food processors. Some persons who have eaten foods containing MSG have reported adverse reactions.

Packaging and examples: The products being recalled are packaged in 121b. cases, each containing four 31b. bags. They are

"Tankersley, Tasty Brand, Food Service, TACO FILLING WITH BEEF AND TEXTURED VEGETABLE PROTEIN, EXCELLENT FILLING FOR TACOS, BURRITOS, TACO SALADS, AND ENCHILADAS."

"Famous Brand, Established in 1935, TACO FILLING WITH BEEF AND TEXTURED VEGETABLE PROTEIN, EXCELLENT FILLING FOR TACOS, BURRITOS AND ENCHILLADAS."

"Quality Foods Inc., TACO FILLING, WITH BEEF AND TEXTURED VEGETABLE PROTEIN."

Fully-cooked, frozen chicken products: Class I Recall (Dec. 12, 2002)

Company: ConAgra Foods Poultry Group, Elberton, Georgia

Date and distribution: The chicken was produced on Aug. 29, 2002. Distribution: Retail stores in Alabama, Florida, Georgia, Illinois, Kentucky, Maryland, Mississippi, Missouri, North Carolina, Tennessee, and Texas.

Product: approximately 36,000 pounds of fully cooked, frozen chicken products

Reason: May be contaminated with a foreign material, plastic

Packaging and examples: Easy Entrée POPCORN STYLE CHICKEN, Fully Cooked, Breaded Chicken Breast with Rib Meat, MADE WITH WHITE MEAT, OVEN CRISP BREADING. The product was packaged in 2 pound bags and bears the code 2241 P184 on the bags.

Frozen, fully-cooked pork dumplings: Class I Recall (Oct. 11, 2002)

Company: Golden Coin Food Industries, Honolulu, Hawaii

Date: Produced on Oct. 2 and distributed to retail establishments on the Hawaiian Islands of Maui and Oahu, in 15 pound cases marked with a case code of 276

Product: 150 pounds of frozen, fully-cooked pork dumplings

Establishment code: EST. 12446

Reason: May be contaminated with *Listeria monocytogenes*. Consumption of food contaminated with *Listeria monocytogenes* can cause listeriosis, an uncommon but potentially fatal disease. Healthy people rarely contract listeriosis. Listeriosis can cause high fever, severe headache, neck stiffness, and nausea. Listeriosis can also cause miscarriages and stillbirths, as well as serious and sometimes fatal infections in those with weak immune systems—infants, the frail or elderly, and persons with chronic disease, with HIV infection, or taking chemotherapy. The USDA/FSIS has received no reports of illnesses associated with consumption of this product.

Package: Golden Coin SHIO MAI PORK HASH DUMPLING FULLY COOKED READY TO SERVE. Packaged in one-pound plastic bags.

Fresh and frozen ground beef products: Class I Recall (Oct. 10, 2002)

Company: EMMPAK Foods Inc., doing business as Peck Meats, Milwaukee, Wisconsin Date: Produced on September 23 and distributed to retail stores and other institutions nationwide

Product: Approximately 568,000 pounds of fresh and frozen ground beef products Establishment code: EST. 20654

Reason: May be contaminated with *E. coli* O157:H7. *E. coli* O157:H7 is a potentially deadly bacterium that can cause bloody diarrhea and dehydration. The very young, seniors, and persons with compromised immune systems are the most susceptible to food-borne illness.

Packaging and examples:

- 1 to 5lb. packages of GROUND BEEF, CONTAINS 8% FAT. Each package also bears one of the following codes: 8659206, 8659204, or 8659200. Each package also bears the sell-by date 10.2.02.
- 1 to 5lb. packages of GROUND BEEF CHUCK PATTIES, CONTAINS 20% FAT. Each package also bears one of the following codes, 8658106 or 8658100. Each package also bears the sell-by date 10.2.02.
- 1 to 5lb. packages of GROUND BEEF ROUND PATTIES, 100% BEEF, CONTAINS 15% FAT. Each package also bears one of the following codes, 8685706, or 8685700. Each package also bears the sell-by date 10.2.02.

Fresh and frozen ground beef products: Class I Recall (Sept. 27, 2002)

Company: EMMPAK Foods Inc., doing business as Peck Meats, Milwaukee, Wisconsin

Date and distribution: Hotels, restaurants and other institutions nationwide

Product: 416,000 pounds of fresh and frozen ground beef products

Establishment code EST. 20654

Reason: May be contaminated with E. coli O157:H7

Packaging and examples:

20 pound boxes of FRESH GROUND BEEF, 2/10, PACKED FOR INSTITUTIONAL USE. 5829940

5 pound bags of Our Own KITCHEN, 20 QUARTER POUND, 100% PURE, BEEF PATTIES.

Ready-to-eat, fresh and frozen pork sausage products: Class I Recall (Dec. 4, 2002)

Company: Crofton and Sons Inc., Tampa, Florida

Date and distribution: Products were produced on Sept. 24. Distribution: Retail stores in Florida

Product: Approximately 8,600 pounds of ready-to-eat, fresh and frozen pork sausage products

Reason: May be contaminated with Listeria monocytogenes

Packaging and examples:

1 pound individually wrapped packages of MILD BEAN BROTHERS Country SMOKED SAUSAGE. Each package is stamped with SELL BY DEC 26.

10 pound cases of Uncle John's Pride Country Smoked Sausage Kielbasa, bearing the code 1350 on the label. Inside each case is a single package of UNCLE JOHN'S PRIDE Kielbasa Smoked Sausage stamped with PACKED ON SEP 27.

Fresh and frozen ready-to-eat turkey: Class I Recall (Oct. 9, 2002)

Company: Pilgrim's Pride Corporation, doing business as Wampler Foods Inc., Franconia, Pennsylvania

Product: Approximately 295,000 pounds of fresh and frozen ready-to-eat turkey and chicken products

Reason: May be contaminated with Listeria monocytogenes

Packaging and examples:

Various sized boxes of WAMPLER FOODS, BONELESS, FULLY COOKED DARK, TURKEY PASTRAMI, 21132.; packed in each box are 4lb. slabs of WAMPLER FOODS, TURKEY PASTRAMI, Fully Cooked, Boneless, Dark. The products subject to recall bear the sell-by date10/8/02.

Various sized boxes of WAMPLER FOODS, OVEN ROASTED-BONELESS, TURKEY BREAST, WITH BROTH, SKINLESS, 11159. Packed in each box are 9 lb. bags of WAMPLER FOODS, TURKEY BREAST, FAT FREE. The products bear the sell-by date of either 11/4/02 and/or 11/5/02.

Frozen salisbury steak products: Class II Recall (Oct. 4, 2002)

Company: Luigino's Inc., Jackson, Ohio

Date and distribution: Produced on April 23 and May 28 and distributed to retail stores in Arizona, Indiana and Wisconsin

Product: Approximately 16,300 pounds

Reasons: Misbranding, including undeclared allergens (eggs). Eggs are known allergens. Persons who have an allergy or severe sensitivity to eggs run the risk of possible allergic reactions if they consume this product.

Packaging and examples:

10.5 ounce boxes of "Michelina's, Signature, SALISBURY STEAK GRAVY, with shells and cheese. The establishment code "EST. 18297" is included on each package. Also embossed on each package is one of the following date codes: "J2113N12" or "J2148N12."

#### IV. IMPORTED SEAFOODS AND ENFORCEMENT INTRODUCTION

The FDA has issued the following guidelines regarding problem imports. The frozen seafood imports head the list of problems.

The information presented in this chapter serves to provide guidance to U.S. federal and state regulators for dealing with importers or other individuals who engage in business practices that appear designed to evade the lawful regulation of imports.

This guidance represents the agency's current thinking on dealing with problem importers. It does not create or confer any rights for or on any person and does not operate to bind the FDA or the public.

Priority attention should be given to firms with a history of any of the following actions:

Distributing imported articles in domestic commerce following receipt of a Notice of FDA Action specifying the intention of Sampling, or the Detention or Refusal of the articles; or prior to receipt of a Notice of FDA Action specifying the articles are Released

Repeatedly importing violative articles

Falsifying documents at time of entry, reconditioning, or reexport, including misdeclaring articles to avoid detention without physical examination or other regulatory action

Reentering previously refused articles into the United States

Failing to recall or redeliver to the U.S. Customs Service, at its request, an article for which a Notice of FDA Action specifying that the article was refused by FDA has been issued

Introducing or delivering for introduction into domestic commerce (after entry) any article that is adulterated or misbranded, or that is a new drug without an approved New Drug Application

Committing any prohibited act (see 21 USC 331)

#### V. IMPORTED SEAFOODS AND ENFORCEMENT BACKGROUND

In developing FDA's automated import system, known as the Operational and Administrative System for Import Support (OASIS), the specific forms "May Proceed Notice," "Release Notice," "Notice of Sampling," "Notice of Detention and Hearing,"

and "Notice of Refusal" have been replaced by the issuing of "Notices of FDA Action," which includes a description of the specific FDA action (May Proceed, Release, Sampling or Intention of Sampling, Detention, or Refusal) identified for the specific line in the entry. The use of the designations "Product May Proceed," "Product Released by FDA," "Product Collected by FDA," "Product Detained by FDA," or "Product Refused Entry by FDA," or similar wording should be considered as meeting the standard, "giving notice thereof to the owner or consignee" [see 21 USC 381(a); 21 CFR 1.94].

In 1988, the Agency conducted a short-term enforcement operation aimed at determining the disposition of food articles refused admission. Thirteen percent of articles refused admission for nonlabeling violations had been distributed in interstate commerce, rather than redelivered for export or destruction.

Between 1990 and 1992, the New York District, in conjunction with the U.S. Customs Service, investigated and documented an importer's history of violative practices regarding the importation of frozen seafood products. Practices included repeatedly importing violative articles; falsifying documents and manipulating articles to avoid detention without physical examination; refusing or not permitting timely inspection of entries; importing previously refused articles; and smuggling. As a result of the investigation, in 1992 the firm's president was indicted by the U.S. District Court in New Jersey. He was subsequently convicted on 138 counts for submitting false documents to the FDA and for illegally reimporting previously rejected salmonella-contaminated seafood. On February 5, 1993, all frozen seafood products imported by the firm were placed on detention without physical examination.

Between 1992 and 1995, the Florida District and the Office of Criminal Investigations, in conjunction with the U.S. Customs Service, investigated and documented an importer's history of violative practices regarding the importation and handling of frozen shrimp. Practices included repeatedly importing violative articles: falsifying documents to avoid detention without physical examination; manipulating articles in attempts to have packers removed from detention without physical examination; and laboratory shopping (sending samples of product that is detained without physical examination to different private labs and then submitting to FDA only the analysis that shows the product in compliance, even though the other laboratory found the product violative). Further, the Florida District identified three shipments of shrimp imported by the firm that were seized because of decomposition. Prior to the seizures, the firm attempted to sell the decomposed shrimp, which had been rejected by eight consignees and the National Marine Fisheries Service. The firm also was discovered washing decomposed imported shrimp with a copper sulfate solution in an attempt to conceal the decomposition. On March 10, 1995, all frozen shrimp imported by the firm was placed on detention without physical examination. As a further result of the investigation, the firm and its top management were indicted by the U.S. District Court in Florida. The firm's vice president was convicted on 12 felony counts, including conspiracy, obstructing justice, violating Customs law, and tainting shrimp and selling it with the intent to defraud and mislead.

The following enforcement approaches have general applicability. They should be considered when dealing with firms engaged in the types of practices listed above, when conventional import coverage and enforcement avenues appear insufficient to address the problem. The approaches include review and approval of reconditioning proposals (FD-766), the use of warning letters (sequential, when appropriate), recall, seizure, injunction, or prosecution.

As always, use of enforcement discretion by the district should be considered in determining the appropriate regulatory response. When egregious actions are encountered, a sequential approach may not be appropriate. Also, situations that appear to involve criminal activity (e.g., smuggling, falsification of records) should be referred to the Office of Criminal Investigations for their information and follow-up, as appropriate.

# A. Warning Letters

Issuance of warning letters to remind firms of their responsibilities to import articles that comply with the provisions of the Federal Food, Drug, and Cosmetic Act and other laws enforced by the FDA, and to assure that only nonviolative articles enter domestic commerce in the United States, is often an appropriate first action. Warning letters may be issued to the importer of record, owner, or consignee (if other than the importer of record) with copies to Customs, and may be issued for the following reasons:

- 1. Failure to hold an entry intact pending receipt of a Notice of FDA Action specifying that the article was released by the FDA. A copy of the warning letter should be attached to the redelivery request sent to Customs when such a request is made.
- 2. The first documented attempted entry with misleading information. Misleading information includes, for example, low-acid canned foods from a nonregistered plant entered under another processor's Food Canning Establishment (FCE) number; or articles from firms subject to detention without physical examination; or articles declared as nonregulated articles to avoid detention without physical examination or other agency action.
- 3. The first documented instance of submission of a foreign government certification document or private laboratory analytical report that does not match the entry in question.
- 4. An importer's failure to provide the FDA with information regarding the availability for sampling or location of an entry for which a Notice of FDA Action specifying the FDA's intention of sampling has been issued.
- 5. To inform an importer that the FDA has requested that Customs deny it permission to file an entry bond, thus restricting its shipments to Customs' custody until admissibility has been determined.
- 6. Consistently importing violative articles not already subject to detention without physical examination. The importer should be notified that this practice may result in future entries being detained without physical examination.
- 7. Any other situation that warrants an official notification to the firm and further opportunity for compliance before other action is taken.

The warning letter should state that any distribution of refused articles or articles sampled or intended for sampling that were distributed prior to release are in violation of the Federal Food, Drug, and Cosmetic Act or other applicable acts enforced by the FDA and may result in domestic seizure or other sanctions, including injunction or prosecution.

#### B. Reconditioning Proposals

The Federal Food, Drug, and Cosmetic Act provides that when an article submitted for entry is found to be violative, the importer has the option of exporting it, destroying it, rendering it not subject to the Act, or requesting permission from the agency to attempt to bring it into compliance with the Act.

If the importer of record decides to attempt to recondition a detained article, Section 801(b) of the Act (21 USC 381 (a)) provides that the owner or consignee (in practice, the FDA also accepts applications from an importer of record, with a properly posted bond, as the agent of the owner or consignee) may submit to the FDA a written application (Form FD-766 or other acceptable means) requesting permission to bring into compliance an article that is adulterated, misbranded, or in violation of Section 505 (see 21 USC 381 (a)(3)). The owner or consignee may bring the article into compliance by relabeling or other action, or by rendering it other than a food, drug, device, or cosmetic.

The approval of the reconditioning application is at the FDA's discretion. The Agency should require appropriate controls and provisions as a part of any application before it approves the reconditioning. The application is an agreement between the importer (or other appropriate party submitting the application) and the agency.

If the FDA has documented an importer's practice of consistently importing violative articles not already subject to detention without physical examination and only attempting to recondition the articles after detention, the District may require, as part of any reconditioning application, that the importer agree to destroy any article not brought into compliance during reconditioning, in lieu of permitting reexport of the violative article.

Districts should consult and obtain the proper authorization before initiating a policy requiring a specific importer to destroy rather than reexport violative articles as part of every reconditioning process. The information supplied should include, but not be limited to, the following:

- 1. Documentation of the firm's pattern of importing violative articles
- 2. Documentation of prior warning to the firm of their obligation to import the article in compliance with the Federal Food, Drug, and Cosmetic Act or other acts enforced by the FDA
- 3. Documentation that may establish that the article can be imported in compliance and thus would not require reconditioning after importation

# C. Requests For Voluntary Recalls

Although requests for voluntary recalls duplicate a request for redelivery action to some degree, they also offer definite advantages. Experience indicates that requesting the firm to initiate a voluntary action, such as a recall, may result in a more favorable response by the firm than a demand for redelivery. A recall may occur more promptly because it can be initiated in a matter of days, while redelivery may not take place for 90 days or more. This is especially significant in hazard-to-health situations. A recall may provide the FDA with further knowledge of the status of the violative merchandise being returned and usually makes it easier to maintain control of the article. This ultimately leads to improved consumer protection.

District management should very carefully encourage the firm to consider a voluntary recall under the following situations:

- 1. When a potential health hazard situation exists
- 2. When there is evidence of distribution of detained or refused merchandise

When an importer fails to respond fully or in a timely manner to a warning letter, or we are notified by Customs that an importer has not responded to a Notice of FDA Action Specifying Refusal of the product, it may be an indication the goods are no longer

intact. A visit to the importer may be appropriate and, if articles are missing, one should attempt to determine the firm's intentions with respect to corrective action.

When a potential health hazard situation exists and the article has been illegally distributed, appropriate press coverage may issue naming firm, product, and country of origin. Issuance of all publicity must be in accordance with guidelines.

Import recalls are to be conducted in full accordance with FDA guidelines. Supervision of the disposition of returned articles may be made either by the FDA or by Customs. If disposition will be by destruction, it is suggested that the FDA provide the supervision. If the articles are to be exported, Customs or the FDA may handle the supervision.

# D. Seizure

Seizure is another enforcement approach that may be considered to gain control over violative imported articles. Seizure is an action against an article. Consequently, it will be necessary to show, through laboratory analysis or otherwise, that the article seized is actually violative. An importer's history of illegal actions, while relevant, is not itself sufficient to support seizure. Whatever the importer's previous history, it will be necessary to show that the article itself is violative. Seizure may be considered for an article which:

- 1. Represents a potential hazard to health and has been or is likely to be distributed in domestic commerce following receipt of a Notice of FDA Action specifying that the article is Detained or Refused
- 2. Has been fraudulently identified/represented in documents submitted to the agency
- 3. Is identified by the agency as a previously refused article.

When an imported article is seized, and condemned, it is subject to the provisions of Section 304(d) (21 USC 334(d)), which may allow for reexportation of the article, provided specified conditions are met.

In order to be able to reexport condemned imported articles, the party seeking reexport must satisfy several threshold conditions:

- 1. The violation did not occur after the article was imported.
- 2. The party seeking reexport "had no cause for believing that it was adulterated, misbranded, or in violation before it was released from Customs custody."
- 3. The party seeking reexport must "establish that the article was intended for export at the time the article entered commerce." An example of where it may be possible to demonstrate that a product was intended for export at the time it entered commerce would be when products are imported for the purpose of transshipment to a destination outside the U.S.
- 4. Compliance with 21 USC 381 (e) (1): Intended for export.
  - (a) Accords with the specifications of the foreign purchaser (unless the article is to be exported to the original foreign supplier, in which case there is no need to comply with this requirement).
  - (b) May not be in conflict with the laws of the country to which it is intended for export (unless the article is to be exported to the original foreign supplier, in which case there is no need to comply with this requirement).
  - (c) Labeled on the outside of the shipping package that it is intended for export.

(d) Not sold or offered for sale in domestic commerce.

Therefore there are circumstances where the seizure of an article may not accomplish more than detention and refusal of the article, other than stricter control over the goods before reexport and compliance with the applicable requirements of Section 801(e) (21 USC 381(e).

Consequently, in evaluating whether a seizure is an appropriate course of action, a district should consider whether the facts in the case would justify recommending to a court that reexport of the article would be an unsatisfactory resolution. Among the points to consider are

- 1. Does a potential health hazard exist?
- 2. Does the previous history of the person in possession of the articles indicate that the person may attempt to reenter the articles into the United States at a later date?
- 3. Did the violation occur after the article was imported?
- 4. Did the importer have cause to believe that the article was in violation before entry?
- 5. Does the article meet the legal specifications of the country to which it would be exported?
- 6. Was any portion of the article sold or offered for sale in domestic commerce?
- 7. Is the article in violation of 21 USC 342(a)(1), (2), or (6), 344, 351(a)(3), 352(j), 355 or 361(a) or (d)?
- 8. If the article is a drug, will it be reexported to the original foreign supplier?

Under certain circumstances, the district may recommend seizure of violative articles under 21 USC 334 while the articles are still under import status, rather than allow reexport as provided under 21 USC 381 (a). Generally, seizure of articles while in import status may be appropriate if the articles must be destroyed (pose a serious health hazard or it is likely that the articles will be reintroduced into the United States) or the public health requires that certain conditions be imposed (e.g., conditions in 21 USC 381(e)(1)).

As with citation, prosecution, and injunction, samples collected for seizure consideration should, whenever possible, include a 702(b) portion (see 21 USC 372 (b)). Such samples should be collected, sealed, analyzed, and otherwise handled in accordance with procedures normally applied to domestic samples.

State embargo authority and Customs holds are alternative methods to gain control over violative articles. Customs may also release an article at our request so that an immediate domestic seizure may be conducted. Moreover, if a violative article represents evidence of a crime, it may be seized pursuant to a criminal search and seizure warrant. These avenues should also be considered, especially if an importer is likely to attempt to quickly reexport the article.

# E. Injunction

If an injunction is the action of choice, the case should be developed in accordance with standard procedures. Injunctions may require a pattern of actual violations with some recognizable danger of a recurrence. The monitoring of an injunction is resource intensive. These facts should be taken into consideration when evaluating this course of action. Also consider that an injunction often results in a hearing more quickly than does a prosecution, particularly if a Temporary Restraining Order (TRO) is requested. This can

result in quick corrective action as well as more rapid and efficient redelivery if this response is requested in the injunction. Also, the burden of proof is less in civil cases than in criminal cases, and an injunction does not preclude subsequent prosecution for the same violation.

When developing an injunction case against an importer or consignee, there must be a well-documented history of an illegal practice. A TRO requires a heightened showing of harm

#### F. Citation/Prosecution

Citation/prosecution should be used when conventional import enforcement approaches are determined to be inadequate to correct violative practices, or the violation is sufficiently egregious to warrant punishment.

Districts should consider the potential impact of developing citation/prosecution recommendations as the action of choice in the following instances:

- 1. Where there is repetitive illegal distribution of articles after issuance of a Notice of FDA Action specifying the intention of sampling or detention
- 2. Where the importer submits false or misleading entry documents
- 3. Where the importer submits false or misleading private laboratory analytical results or false certifications
- 4. Where the importer submits false or misleading export documents
- 5. Where the importer repeatedly brings previously refused articles into the United States
- 6. Where evidence of other fraud exists

This list is not all-inclusive, and there may be other situations where citation/prosecution is appropriate.

Any recommendation for citation, prosecution, or injunction must be supported by fully documented instances of attempts to circumvent normal import procedures. For a felony prosecution recommendation, there must be a fully documented attempt to do the same, with evidence of the intent to defraud or mislead. It is not necessary, in developing a citation/prosecution recommendation, to show that each specific entry is actually violative. However, physical evidence that documents the violative nature of an entry (or of several entries) would be useful to highlight the likely result of the firm's pattern of behavior.

It is important to remember that sample collection and analytical procedures in these cases, as for seizures and injunctions, should differ from routine import work. When an import physical sample is collected for use in an anticipated legal action, a sealed 702(b) portion should be available (21 USC 372 (b). A proper chain of custody should also be maintained for these samples. Ordinarily, check analyses should be conducted on such samples.

Importers of articles detained without physical examination should not feel free to distribute and sell such articles without risk of criminal penalty. Criminal action may be possible against importers who violate the FDA's detention without physical examination actions or who routinely ship articles without a Notice of FDA Action indicating the articles are released. Refusal to allow inspection is a violation of the Federal Food, Drug, and Cosmetic Act. Subsequent entry pursuant to an inspection warrant may yield evidence providing the basis for a felony violation for refusal to allow inspection. Distribution of an article prior to receipt of a Notice of FDA Action indicating the article may proceed or is

released should be considered refusal to permit inspection, as authorized by section 704 (21 USC 374).

In addition to charges under the Federal Food, Drug, and Cosmetic Act and Customs law, Title 19 (note especially, 19 USC 1592 and 1595a), and/or Title 18, other charges may also be considered. These include 18 USC 1001, false statements; 18 USC 1505, obstruction of justice (when a firm knowingly and willingly interferes with an FDA inspection by distributing imported articles not released by FDA from import status); 18 USC 542, entry by use of a false statement; 18 USC 545, smuggling; and 18 USC 371, conspiracy.

#### **ACKNOWLEDGEMENT**

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Appendix A

FDĀ Standard for Frozen Vegetables: 21 CFR 158. Definitions: 21 CFR 158.3; FDA Standard for Frozen Vegetables: 21 CFR 158. Frozen

peas: 21 CFR 158.170

# I. FDA STANDARD FOR FROZEN VEGETABLES: 21 CFR 158. DEFINITIONS: 21 CFR 158.3

For the purposes of this part the following definitions shall apply:

- (a) Lot. A collection of primary containers or units of the same size, type and style manufactured or packed under similar conditions and handled as a single unit of trade.
- (b) Lot size. The number of primary containers or units (pounds when in bulk) in the lot.
  - (c) Sample size. The total number of sample units drawn for examination from a lot.
- (d) Sample unit. A container, a portion of the contents of a container, or a composite mixture of product from small containers that is sufficient for the examination or testing as a single unit.
- (e) Defective. Any sample unit shall be regarded as defective when the sample unit does not meet the criteria set forth in the standards.
- (f) Acceptance number. The maximum number of defective sample units permitted in the sample in order to consider the lot as meeting the specified requirements. The following acceptance numbers shall apply:

	Size container	
Lot size (primary container)	n <sup>a</sup>	c <sup>b</sup>
Net weight equal to or less than	1 kg (2.2 lb)	(lb)
4,800 or less	13	2
4,801 to 24,000	21	3
24,001 to 48,000	29	4
48,001 to 84,000	48	6
84,001 to 144,000	84	9
144,001 to 240,000	126	13
Over 240,000	200	19

	Size container	
Lot size (primary container)	n <sup>a</sup>	c <sup>b</sup>
Net weight greater than 1 kg	(2.21b) (lb)	
20,000 or less	13	2
More than 20,000 to 100,000	21	3
More than 100,000 to 200,000	29	4
More than 200,000 to 400,000	48	6
More than 400,000 to 600,000	84	9
More than 600,000 to 1,000,000	126	13
More than 1,000,000	200	19

<sup>&</sup>lt;sup>a</sup> n = number of sample units.

(g) Acceptable quality level (AQL). The maximum percentage of defective sample units permitted in a lot that will be accepted approximately 95% of the time.

# II. FDA STANDARD FOR FROZEN VEGETABLES: 21 CFR 158. FROZEN PEAS: 21CFR 158.170

- (a) Identity—(1) Product definition. Frozen peas is the food in "package" form as that term is defined in Sec. 1.20 of this chapter, prepared from the succulent seed of the pea plant of the species *Pisum sativum* L. Any suitable variety of pea may be used. It is blanched, drained, and preserved by freezing in such a way that the range of temperature of maximum crystallization is passed quickly. The freezing process shall not be regarded as complete until the product temperature has reached  $-18^{\circ}$ C (0°F) or lower at the thermal center, after thermal stabilization. Such food may contain one, or any combination of two or more, of the following safe and suitable optional ingredients:
  - (i) Natural and artificial flavors.
  - (ii) Condiments such as spices and mint leaves.
  - (iii) Dry nutritive carbohydrate sweeteners.
  - (iv) Salt.
  - (v) Monosodium glutamate and other glutamic acid salts.
- (2) Size specifications. If size graded, frozen peas shall contain not less than 80% by weight of peas of the size declared or of smaller sizes. The sample unit may not contain more than 20% by weight of peas of the next two larger sizes, of which not more than one quarter by weight of such peas may be of the larger of these two sizes, and may contain no peas larger than the next two larger sizes, if such there be. The following sizes and designations shall apply:

<sup>&</sup>lt;sup>b</sup> c = acceptance number.

	Round hole sieve size through which peas will pas		
Size designation	Millimeters	Inch	
Extra small	Up to 7.5	0.295	
Very small	Up to 8.2	0.32	
Small	Up to 8.75	0.34	
Medium	Up to 10.2	0.40	
Large	Over 10.2	0.40	

- (3) Labeling. The name of the product is "peas". The term "early," "June," or "early June" shall precede or follow the name in the case of smooth-skin or substantially smooth-skin peas, such as Alaska-type peas. Where the peas are of sweet green wrinkled varieties, the name may include the designation "sweet," "green," "wrinkled," or any combination thereof. The label shall contain the words "frozen" or "quick frozen." The name of the food shall include a declaration of any flavoring that characterizes the product as specified in Sec. 101.22 of this chapter and a declaration of any condiment such as spices and mint leaves that characterizes the product, e.g., "Spice added." Where a statement of pea size is made, such statement shall indicate either the size designation as specified in paragraph (a) (2) of this section or the applicable sieve size. However, the optional descriptive words "petite" or "tiny" may be used in conjunction with the product name when an average of 80% or more of the peas will pass through a circular opening of a diameter of 8.75 mm (0.34 in.) or less for sweet green wrinkled peas and 8.2 mm (0.32 in.) for smooth-skin or substantially smooth-skin peas, such as Alaska-type peas.
- (4) Label declaration. Each of the ingredients used in the food shall be declared on the label as required by the applicable sections of parts 101 and 130 of this chapter.
  - (b) Quality. (1) The standard of quality for frozen peas is as follows:
  - (i) Not more than 4% by weight blond peas, i.e., yellow or white but edible peas;
- (ii) Not more than 10% by weight blemished peas, i.e., slightly stained or spotted peas;
- (iii) Not more than 2% by weight seriously blemished peas, i.e., peas that are hard, shrivelled, spotted, discolored or otherwise blemished to an extent that the appearance or eating quality is seriously affected.
- (iv) Not more than 15% by weight pea fragments, i.e., portions of peas, separated or individual cotyledons, crushed, partial or broken cotyledons and loose skins, but excluding entire intact peas with skins detached;
- (v) Not more than 0.5% by weight, or more than 12 sq cm (2 sq in.) in area, extraneous vegetable material, i.e., vine or leaf or pod material from the pea plant or other such material per sample unit as defined in paragraph (b) of this section.
- (vi) The sum of the pea material described in paragraphs (b) (1) (i), (ii), (iii), and (iv) of this section shall not exceed 15%.
- (vii) For peas that meet the organoleptic and analytical characteristics of sweet green wrinkled varieties:
- (a) The alcohol-insoluble solids may not be more than 19% based on the procedure set forth in paragraph (b) (3) of this section.
- (b) Not more than 15% by count of the peas may sink in a solution containing 16% salt by weight according to the brine flotation test set forth in paragraph (b) (4) of this section;

- (viii) For smooth-skin or substantially smooth-skin varieties the alcohol-insoluble solids may not be more than 23% based on the procedure set forth in paragraph (b) (3) of this section.
- (ix) The quality of a lot shall be considered acceptable when the number of defectives does not exceed the acceptance number in the sampling plans set forth in Sec. 158.3 (f).
- (2) The sample unit for determining compliance with the requirements of paragraph (b) (1) of this section other than those of paragraphs (b) (1) (vii) (a) and (b) (1) (viii) of this section, shall be 500 g (17.6 oz). For the determination of alcohol-insoluble solids as specified in paragraph (b) (3) of this section, the container may be the sample unit.
  - (3) Alcohol-insoluble solids determination. (i) Extracting solutions:
- (a) One hundred parts of ethanol denatured with five parts of methanol volume to volume (formula 3A denatured alcohol), or
- (b) A mixture of 95 parts of formula 3A denatured alcohol and five parts of isopropanol v/v.
- (ii) Eighty% alcohol (8 liters of extracting solutions, specified in paragraph (b) (3) (i) (a) or (b) of this section, diluted to 9.5 liters with water).
  - (iii) Drying dish—a flat-bottom dish with a tight fitting cover.
- (iv) Drying oven—a properly ventilated oven thermostatically controlled at  $100 \pm 2^{\circ}\mathrm{C}$
- (v) Procedure—Transfer frozen contents of package to plastic bag; tie bag securely and immerse in water bath with continuous flow at room temperature. Avoid agitation of bag during thawing by using clamps or weights. When sample completely thaws, remove bag, blot off adhering water, and transfer peas to U.S. No. 8 sieve, using (20 cm) size for container of less than 3 lb net weight and (30.5 cm) for larger quantities. Without shifting peas, incline sieve to aid drainage, drain 2 minutes. With cloth wipe surplus water from lower screen surface. Weigh 250 g of peas into high-speed blender, add 250 g of water and blend to smooth paste. For less than 250 g sample, use entire sample with equal weight of water. Weight 20 g + 10 mg of the paste into 250 mL distillation flask, add 120 mL of extracting solutions specified in paragraph (b) (3) (i) (a) or (b) of this section, and reflux 30 minutes on steam or water bath or hotplate. Fit into a buchner funnel a filter paper of appropriate size (previously prepared by drying in flat-bottom dish for 2 hours in drying oven, covering, cooling in desiccator, and weighing). Apply vacuum to buchner funnel and transfer contents of beaker so as to avoid running over edge of paper. Aspirate to dryness and wash material on filter with 80% alcohol until washings are clear and colorless. Transfer paper and alcohol-insoluble solids to drying dish used to prepare paper, dry uncovered for 2 hours in drying oven, cover, cool in desiccator, and weight at once. From this weight deduct weight of dish, cover, and paper. Calculate% by weight of alcoholinsoluble solids.
- (4) Brine flotation test. (i) Explanation—The brine flotation test utilizes salt solutions of various specific gravities to separate the peas according to maturity. The brine solutions are based on the percentage by weight of pure salt (NaCl) in solution at 20°C. In making the test the brine solutions are standardized to the proper specific gravity equivalent to the specified "percent of salt solutions at 20°C" by using a salometer spindle accurately calibrated at 20°C. A 250 mL glass beaker or similar receptacle is filled with the brine solution to a depth of approximately 50 mm. The brine solution and sample (100 peas per container) must be at the same temperature and should closely approximate 20°C.
- (ii) Procedure—After carefully removing the skins from the peas, place the peas into the solution. Pieces of peas and loose skins should not be used in making the brine flotation test. If cotyledons divide, use both cotyledons in the test and consider the two

separated cotyledons as 1 pea; and, if an odd cotyledon sinks, consider it as one pea. Only peas that sink to the bottom of the receptacle within 10 seconds after immersion are counted as "peas that sink."

(5) If the quality of the frozen peas falls below the standard prescribed in paragraph (b) (1) of this section, the label shall bear the general statement of substandard quality specified in the Code of Federal regulations but in lieu of the words prescribed in the second line of the rectangle the following words may be used where the frozen peas fall below the standard in only one respect: "Below standard in quality\_\_\_\_\_," the blank to be filled in with the specific reason for substandard quality as listed in the standard.

# **Appendix B**

# Frozen Dessert Processing: Quality, Safety, and Risk Analysis. Special Operations

#### **GENERAL INSTRUCTIONS**

These guidelines provide recommendations to make an assessment of the safety and quality of frozen desserts from raw ingredients to packaged finished products and set action priority recommendations.

An action priority provides guidance as to what to do first in responding to product safety problems.

#### Risk Assessment

Throughout these processing guidelines, each item or area has been assigned a suggested risk assessment: (H) high risk, (M) moderate risk, or (L) low risk. These are suggested risk assessments. These are general assessments and may not represent specific individual circumstances. If an observed condition constitutes a risk higher or lower than that suggested in these guidelines, the corresponding Action Priority would apply.

#### **IMPORTANT**

The Risk is automatically "H" or "High" when the problem observed is a critical processing element involving

- 1. Proper pasteurization, whereby every particle of milk, milk product, or mix may not have been heated to the proper temperature and held for the required time in properly designed and operated equipment
- 2. A cross-connection whereby direct contamination of milk, milk products, or mix is occurring
- 3. Conditions whereby direct contamination of pasteurized product is occurring

#### The Action Priorities

The three risk categories are defined in terms of appropriate monitoring levels and action priority:

(H) High Risk: High level of control needed because of immediate impact on

product safety. Potential for a problem is high without appropriate

monitoring.

Action Priority—No product should be processed until the problem is corrected. Product on hand should be checked for contamination if appropriate. If product on hand is found to be contaminated, appropriate action should be taken.

(M) *Moderate Risk*:

Potential for a problem is somewhat limited to abuse or particular criteria. Timely monitoring is required because problems in these areas could result in a risk to product safety.

Action Priority—Correction of these problems is necessary within a short period of time. A few days or weeks may be reasonable. Specific additional monitoring is needed until the correction has been accomplished.

(L) Low Risk:

Monitoring needed only on inspection or random-checking basis. Risk potential is low, and significant risk would only result from extensive abuse or extenuating circumstances.

Action Priority—Correction is necessary to help assure ultimate product safety. However, the time frame for correction can be flexible and based around nonpublic health issues such as production schedules. Until the correction is accomplished, routine checks should be made to provide assurance that the status has not changed to "M" or "H".

The action priorities in these guidelines were formulated to be compatible with a Hazard Analysis Critical Control Point (HACCP) system. However, in order to implement a full HACCP program, individual in plant monitoring points and frequencies should be established.

#### **SPECIFIC OPERATIONS**

#### Receiving

*Note:* Some plants receive pasteurized products for further processing. Regardless of the specific products received, for discussion purposes, they will be considered as "raw milk" products.

*Tanks*—(washing of outside)

Risk L Tankers can be a source of contamination; the exterior of the tanker or tractor should not be washed in the receiving bay. The only exceptions are the valve, hoses, and other parts inside the rear doors of the tanker and the outside of the rear-door area.

#### Drivers

Risk M Because drivers work in and around farms during pickups where the possibility exists of their coming in contact with contamination, there is a possibility of their introducing contamination in a plant if their access is not limited.

Drivers should not be allowed in processing areas under any circumstances. Separate toilet and hand washing facilities should be made available to them.

#### Receiver

Risk M Like the drivers, the receiving person has direct contact with the raw product. This person should be isolated from the rest of the plant when working, and access to the receiving area by other personnel should be strictly limited. The receiving person should have a telephone or intercom for communications and should wear an outer garment of distinctive color, a different color from the rest of the employees' garments. When the receiving person leaves the receiving area, he/she should shed his/her outer garment and then clean and sanitize his/her boots and hands. This person should be trained to examine incoming raw product.

#### Tank Truck Unloading Practices

Risk L The product in tank trucks should be protected during unloading; therefore products in tank trucks should only be transferred or received if the area is completely enclosed (walls and ceiling, with doors closed) during the unloading process and the dust cover or dome is closed. If this is the case, the manhole cover can be opened slightly and held in this position by the metal clamps used to close the cover; and a filter is not needed. If the area is not enclosed or doors of the unloading area are open during unloading, a suitable filter should be used for the manhole or air Inlet vent and suitable protection provided over the filter material either by design of the filter-holding apparatus or a roof or ceiling over the area.

Direct connections to the milk tank truck should be made to the valve or ferrule to-ferrule through the manhole lid. Adequate protection should be provided for the air vent.

#### Receiving Room Construction and Cleaning

Risk L Floors, drains, walls, and ceilings, etc. need to be kept clean and in good repair.

# Sample Ladle

Risk L The ladle, or the device that is used to take the sample, should be kept in a sanitizing solution when it is not in use. This solution should be changed as needed throughout the day. Automatic samplers should be cleaned after each use.

# Tanker Agitator

Risk L The tanker agitator should be cleaned and sanitized after each use. The agitator should be free of cracks or crevices where product residues can form.

If direct air agitation is used, air should be clean, dry, and oil free, and filters should be employed and changed daily. Mechanical agitators should be constructed to adequately cover the manhole opening and protect the product in the truck during agitation.

# Sampling Station

Risk L Samples should be taken quickly using a sterile sampling bag. Samples should be delivered to the laboratory without having to enter the processing area.

## Traceability

Risk M In case of a problem, the sources of dairy products received should be traceable. Also, knowledge of where and when the tank truck was last cleaned and sanitized needs to be available before the products are unloaded.

#### Receiving Hose and Lines

Risk L The receiving hose should be durable and in good repair. After cleaning and sanitizing, lines and hoses should be capped and stored off the floor. Provisions are needed for adequate cleaning and sanitizing of all milk hoses, including the farm pickup hose. Pipelines should be self-draining and in good repair.

# **Raw Product Handling and Mix Preparation**

Room Construction and Cleaning

Risk L Floors, drains, walls, and ceilings, etc. need to be kept clean and in good repair.

## Storage Tanks and Silos

- Risk M Each tank or silo should be cleaned each time it is emptied. Products should be held no longer than three days prior to pasteurization. During each cleaning, the door, gasket, mechanical agitator, petcock, and valve should be removed and cleaned by hand.
- Risk L Air to vent tanks and silos should be drawn from a clean, dry area similar to a processing area. Air from truck bays or other relatively unprotected areas should only be used if it is drawn through a properly designed and operating filter.

# Blending Frozen Dessert Mixes

- Risk H Dusty, raw ingredient blending operations that create powdery conditions should be located away from pasteurized product areas.
- Risk M Blending and mix preparation which exposes the ingredients should be carried out in the processing room.

Except when ingredients are being added, all openings into vessels and lines containing product need to be kept covered and/or capped. Fill lines entering tanks and vats should be connected to the valve or through properly protected opening in the lid. A tank opening should not be held or blocked open by the fill line.

Risk M If powdered ingredients are to be dumped into a vessel for mixing, the outer box or wrapper should be removed before the contents are dumped.

# Cooling

Risk M All liquid ingredients that will support bacterial growth need to be kept or immediately cooled to 45°F, or below.

#### **Pasteurization**

Risk M–H. (H applies if the problem results in underprocessed product). APPLICABLE TO ALL SUBSECTIONS IN THE PASTEURIZATION SECTION OF THESE GUIDELINES.

With the exception of low-acid canned food processes, pasteurization is the only acceptable, practical, commercial measure that if properly applied to all milk, milk products, and mix, will destroy milk-borne disease organisms. Therefore all frozen dessert mixes, dairy and nondairy, need to be pasteurized.

A note of caution is in order. Although pasteurization devitalizes organisms, it does not destroy toxins that may be formed in ingredients or frozen dessert mix when certain staphylococci organisms are present (as from udder infections) and when the milk or mix is not properly refrigerated before pasteurization. Such toxins may cause severe illness.

Properly applied pasteurization assures that every particle of milk or milk products including frozen dessert mixes are heated to at least a minimum temperature and held at that temperature for at least the specified time in properly designed, installed, and operated equipment.

Minimum Pasteurization Times and Temperatures for Milk and Milk Products Are

Milk	145°F 161°F 191°F 194°F 201°F	30 minutes 15 seconds 1 second 0.5 second	
Milk Products of 10%	204°F 212°F 150°F	0.05 second 0.01 second 30 minutes	
fat or more or with added sweeteners, i.e., chocolate milk, cream, etc.	166°F 191°F 194°F 201°F 204°F 212°F	15 seconds 1 second 0.5 second 0.1 second 0.05 second 0.01 second	

Minimum Times and Temperatures for Frozen Dessert Mixes

Temperature	Time
155°F	30 minutes
175°F	25 seconds
180°F	15 seconds
191°F	1.0 second
194°F	0.5 second
201°F	0.1 second
204°F	0.05 second
212°F	0.01 second

Other minimum times and temperatures may be used only if they have been recognized to be equally effective by the Food and Drug Administration and approved by the regulatory agency.

It is recommended that the minimum pasteurization time/temperature combinations be exceeded where possible.

In the next sections, the acceptable types of pasteurization, vat, high temperature short time (HTST), and higher heat shorter time (HHST) are discussed.

Any other equipment, design, layout, testing method, or operational practice can be used and meets the requirements of this guideline if it is acceptable to the U.S. Food and Drug Administration and the local regulatory agency.

# **Batch (Vat) Pasteurization**

Vat pasteurization should be performed in equipment that is properly designed, installed, and operated and that insures that every particle of frozen dessert mix, milk, or milk product being pasteurized has been held continuously at or above the proper temperature for at least the specified period of time.

Valves and connections should be properly designed to prevent pockets of cold product within the system. Outlet valves should be inspected regularly to detect leaking and should be of a leak-detection type.

Foam, which is an excellent insulator, should be minimized in the vat during filling, heating, and holding. Covers should remain in place at all times while the product is in the vat.

The airspace between the product and the top of the vat should be maintained at 5°F above minimum pasteurization temperatures. This is necessary to assure that any product, including foam, reaches proper pasteurization temperatures. Reliable and accurate recording, indicating, and airspace thermometers should be present and functioning properly.

#### Time and Temperature Controls

## 1. Temperature Difference

The pasteurizer should be so designed that the simultaneous temperature difference between the warmest and the coldest product in the vat will not exceed 1°F at any time during the holding period.

The vat should be provided with adequate agitation operating throughout the holding period. No batch of frozen dessert mix should be pasteurized unless it covers a sufficient area of the agitator to insure adequate agitation.

2. Location and Required Readings of Indicating and Recording Thermometers Each batch pasteurizer should be equipped with both an indicating and a recording thermometer.

The thermometers should read not less than the required pasteurization temperature throughout the required holding period. The plant operator should check the temperature shown by the recording thermometer against the temperature shown by the indicating thermometer daily; this comparison should be noted on the recording thermometer chart. The recording thermometer should not read higher than the indicating thermometer. No batch of frozen dessert mix should be pasteurized unless it is sufficient to cover the bulbs of both the indicating and the recording thermometer.

Assurance of Minimum Holding Periods—Batch pasteurizers should be so operated that every particle of frozen dessert mix will be held at not less than the minimum pasteurization temperatures continuously for at least 30 minutes. When frozen dessert mix is raised to pasteurization temperature in the vat and cooling is begun in the vat before the opening of the outlet valve, the recorder chart should show at least 30 minutes at not less than minimum pasteurization temperature. When frozen dessert mix is preheated to pasteurization temperature before entering the vat, the recorder chart should show a holding period of at least 30 minutes at not less than the minimum pasteurization temperature plus the time of filling from the level of the recorder bulb. When cooling is begun in the holder after the opening of the outlet valve or is done entirely outside the holder, the chart should show at least 30 minutes at not less than minimum pasteurization temperature plus the time of emptying to the level of the recording thermometer bulb.

When the recorder time interval on the recorder chart at the pasteurization temperature includes filling and/or emptying time, such intervals should be indicated on the recorder chart by the operator by removing the recording thermometer bulb from the milk for a sufficient time to depress the pen, or by turning cold water into the vat jacket at the end of the holding period or by inscribing the holding time on the chart. The filling time and the emptying time for each vat so operated should be determined by the regulatory agency initially and after any change that may affect these times.

No product should be added to the vat after the start of the holding period.

3. Recording Thermometers for Batch Pasteurizers

Utilizing Temperatures Less Than 160°F

Case—Moisture proof under normal operating conditions in pasteurization plants.

Scale—Should have a span of not less than 20°F including pasteurization temperature, plus or minus 5°F graduated in temperature scale divisions of 1°F spaced not less than 0.0625 of an inch apart between 140°F and 155°F. Provided that temperature scale divisions of 1°F spaced not less than 0.040 of an inch apart are permitted when the ink line is thin enough to be easily distinguished from the printed line, graduated in time scale divisions of not more than 10 minutes, having a cord of straight-line length of not less than 0.25 of an inch between 145°F and 150°F.

Temperature Accuracy—Within 1°F, plus or minus, between 140°F and 155°F.

Time Accuracy—The recorded elapsed time, as indicated by the chart rotation, should not exceed the true elapsed time, as compared to an accurate watch over a period of at least 30 minutes at pasteurization temperature. Recorders for batch pasteurizers may be equipped with spring operated or electrically operated clocks.

Pen-Arm Setting Device—Easily accessible; simple to adjust.

Pen and Chart Paper—Pen designed to give line not over 0.025 of an inch wide; easy to maintain.

Submerged Stem Fitting—Pressure-tight seat against inside wall of holder, no threads exposed to product. Distance from underside of ferrule to the sensitive portion of the bulb to be not less than 3 inches.

Chart Speed—A circular chart should make one revolution in not more than 12 hours. Two charts should be used if operations extend beyond 12 hours in one day. Circular charts should be graduated for a maximum record of 12 hours. Strip charts may show a continuous recording over a 24 hour period.

Chart Support Drive—The rotating chart support drive should be provided with a pin to puncture the chart in a manner to prevent its fraudulent rotation.

Utilizing Temperatures 160°F and Above

Batch pasteurizers used solely for 30 minute pasteurization of frozen dessert mix at temperature above 160°F may use recording thermometers with the following options:

Scale—graduated in temperature scale divisions of 2°F spaced not less than 0.040 of an inch apart between 150°F and 170°F, and graduated in time scale divisions of not more than 15 minutes, having a cord of straight line length of not less than 0.25 of an inch between 160°F and 170°F.

Temperature Accuracy—Within 2°F, plus or minus, between 160°F and 170°F. Chart Speed—A circular chart should make one revolution in not more than 24 hours, and should be graduated for a maximum record of 24 hours.

4. Indicating Thermometers for Batch Pasteurizers

Mercury-actuated, direct-reading; contained in a corrosion-resistant case that protects against breakage and permits easy observation of column and scale filling above mercury, nitrogen, or other suitable gas.

Magnification of Mercury Column—To apparent width of not less than 0.0625 of an inch.

Scale—Should have a span of not less than 25°F including the pasteurization temperature plus and minus 5°F; graduated in 1°F divisions with not more than 16°F per inch of span; protected against damage at 220°F.

Accuracy—Within 0.5°F, plus or minus, through the specified scale span.

Submerged Stem Fitting—Pressure-tight seat against inside wall of holder; no threads exposed to product location of seat to conform to that of a 3-A Sanitary Standard wall type fitting or other equivalent sanitary fitting.

Bulb—Corning normal or equally suitable thermometric glass.

*Note*: On vat pasteurizers used solely for temperatures above 160°F, indicating thermometers with a scale not more than 28°F per inch and 2°F graduations may be used. These thermometers should be accurate to within 1°F, plus or minus, throughout the specified scale range.

- 5. Airspace Heating
- a. Means should be provided and used in batch pasteurizers to keep the atmosphere above the frozen dessert mix at a temperature not less than 5°F higher than the minimum required temperature of pasteurization during the holding period.
- b. Each batch pasteurizer should be equipped with an airspace thermometer. The surface of the frozen dessert mix should be at least one inch below the bottom of the thermometer bulb when the vat is in operation.
- c. The temperature shown by the airspace thermometer should be recorded on the recording thermometer chart each time the pasteurizer is in operation.

6. Airspace indicating Thermometer for Batch Pasteurizers

Type—Mercury-actuated direct-reading; contained in corrosion-resistant case that protects against breakage and permits easy observation of column and scale; bottom of bulb chamber not less than two inches and not more than 3.5 inches below underside of cover; filling above mercury, nitrogen, or equally suitable gas.

Magnification of Mercury Column—To apparent width of not less than 0.0625 of an inch.

Scale—Should have a span of not less than 25°F, including 150°F, plus or minus 5°F; graduated in not more than 2°F divisions, with not more than 16°F per inch of scale; protected against damage at 220°F.

Accuracy—Within 1°F, plus or minus, through the specified scale span.

Stem Fittings—Pressure-tight seat or other suitable sanitary fittings. No threads exposed.

Inlet and Outlet Valves and Connections

#### **Definitions**

- 1. "Valve Stop" should mean a guide that permits turning the valve plug to, but not beyond, the fully closed position.
- 2. "90 stop" should mean a stop so designed as to prevent turning the plug more than  $90^{\circ}$ .
  - 3. "120° stop" should mean a stop which prevents turning the plug more than 120°.
- 4. " $180^{\circ}$  stop" should mean a stop which prevents turning the plug more than  $180^{\circ}$ , but which permits two fully closed positions, each diametrically opposite each other.
- 5. "Valve with an irreversible plug" should mean one in which the plug cannot be reversed in the shell.
- 6. "Single-quadrant stop" should mean a  $90^{\circ}$  stop in a valve with an irreversible plug.
- 7. "The fully open position" should mean that position of the valve seat which permits the maximum flow into and out of the pasteurizer.
- 8. "The closed position" should mean any position of the valve seat that stops the flow of frozen dessert mix into or out of the pasteurizer.
- 9. "The fully closed position" should mean that closed position of the valve seat that requires the maximum movement of the valve to reach the fully open position.
- 10. "The just-closed position" should mean that closed position of a plug-type valve in which the flow into or out of the holder is barely stopped, or any closed position within 0.078 of an inch thereof as measured along the maximum circumference of the valve seat.
- 11. "Leakage" should mean the entrance of unpasteurized frozen dessert mix into a batch pasteurizer during the holding or emptying period, or the entrance of unpasteurized frozen dessert mix into any pasteurized frozen dessert mix line at any time.
- 12. "Leak-protector valve" should mean a valve provided with a leak diverting device that, when the valve is in any closed position, will prevent leakage of frozen dessert mix past the valve.
- 13. "Closed-coupled valve" should mean a valve, the seat of which is either flush with the inner wall of the pasteurizer or so closely coupled that no frozen dessert mix in the valve inlet is more than 1°F colder than the mix at the center of the pasteurizer at any time during the holding period.

A closed-coupled valve that is not truly flush should be considered as satisfying this requirement when all of the following are true:

- a. The vat outlet is so flared that the smallest diameter of the large end of the flare is not less than the diameter of the outlet line, plus the depth of the flare.
- b. The greatest distance from the valve seat to the small end of the flare is not greater than the diameter of the outlet line.
- c. In the case of batch pasteurizers, the outlet and the agitator are so placed as to insure that currents will be swept into the outlet.

# Design and Installation

- 1. Valves and pipeline connections should meet the equipment construction and repair sections of this document.
- 2. All pipelines and fittings should be so constructed and so located that leakage will not occur. Dependence should not be placed on soldered joints to prevent leakage.
- 3. To prevent clogging and to promote drainage, all leak-protection grooves should be at least 0.187 of an inch wide, and at least 0.094 of an inch deep at the center. Mating grooves should provide these dimensions throughout their combined length whenever the valve is in, or approximately in, the fully closed position. All single-leak grooves and all mating leak grooves when mated should extend throughout the entire depth of the seat. Washers or other parts should not obstruct leak-protector grooves.
- 4. A stop should be provided on all plug-type outlet valves and on all plug-type inlet valves in order to guide the operator in closing the valve so mix may not inadvertently be permitted to enter the outlet line or the holder, respectively. In the case of three-way plug-type valves (i.e., those having only one inlet and one outlet), a 180 degree stop, or any combination of stops permitting two fully closed positions, may be submitted for a 90 degree stop, provided that there are no air-relief grooves in the plug and that all leak grooves are located symmetrically with respect to the valve inlet. Stops should be so designed that the operator cannot turn the valve beyond the stop position, either by raising the plug or by any other means.
- 5. Outlet valves, in addition to the requirements listed above, should be so designed as to prevent the accumulation of frozen dessert mix in the product passages of the valve when the valve is in any closed position.
- 6. All inlet pipelines and outlets from vat pasteurizers should be equipped with leak-protector valves, provided that installations not equipped with leak-protector inlet valves should be accepted when the piping is so arranged that only one vat can be connected to the inlet line at a time, and such piping is disconnected during the holding and emptying periods.
- 7. Inlet and outlet connections other than through close-coupled valves should not enter nor leave the pasteurizer below the level of the frozen dessert mix therein.
- 8. In cases where the inlet line enters the holder above the product level, and in which the inlet line may be submerged and thus prevent its complete emptying when the inlet valve is closed, the inlet line should be provided with an automatic air-relief or vent located either at the valve or elsewhere, and so designed as to function in every closed position of the valve. A vent may be provided by drilling a hole at least 0.125 of an inch in diameter in the vat pipe, below the vat cover, but above the maximum product level.
- 9. All leak-protector valves should be installed in the proper position to insure the function of the leak-diverting device. Inlet should not be located in vertical pipelines,

unless they can be so installed that one of the groove systems is at the lowest level of the valve; pipelines between the inlet valve and the pasteurizer are as short as practicable and sloped to drain.

10. All outlet valves should be kept fully closed during filling, heating, and holding periods; and all inlet valves should be kept fully closed during holding and emptying periods.

## Temperature Recording Charts

All temperature recording charts should be preserved for a minimum period of three months or from the time of the last regulatory inspection, whichever is longer. Because of the great value of these records in determining proper pasteurization after the fact, those charts should be preserved for at least as long as the shelf life of the finished frozen dessert.

The use of such charts should not exceed the time limit for which they are designed. There should be no overlapping of recorded data. The following information should be entered on the charts, as applicable:

- 1. Date
- 2. Number or location of recorder when more than one are used
- 3. Extent of holding period, including filling and emptying times when required
- 4. Reading of airspace thermometer within the holding period at a given time or reference point as indicated on the chart
- 5. Reading of indicating thermometer within the holding period at a given time or reference point as indicated on the chart
- 6. Quarterly, the initials of the regulatory agency or plant quality control person opposite the required readings of the indicating thermometer and airspace thermometer
- 7. Quarterly, the time accuracy of the recorder, as determined by the regulatory agency or plant quality control person
- 8. Amount and name of pasteurized frozen dessert mix represented by each batch or run on the chart
- 9. Record of unusual occurrences
- 10. Signature or initials of operator
- 11. Name of plant

## Required Tests

The following tests should be performed by a regulatory agency and deviations corrected.

- Test 1 Indicating thermometer—temperature accuracy (applies to indicating and airspace thermometers)
- Test 2 Recording thermometer—temperature accuracy
- Test 3 Recording thermometer—time accuracy
- Test 4 \*Recording thermometer check against indicator
- Test 5 Leak protector valve

*Note*: Appropriate procedures for these tests appear in the latest edition of the "Grade A Pasteurized Milk Ordinance."

<sup>\*</sup> Also should be done daily by the operator.

# High Temperature Short-Time (HTST) and Higher Heat Shorter Time (HHST) Continuous Flow Pasteurization

In order to assure pasteurization, the following specifications and requirements need to be met:

Automatic Controls—Each high-temperature, short-time, continuous flow pasteurization system should be equipped with an automatic product flow control of the diversion type that complies with the following definition, specifications, and performance requirements:

The term "automatic controls" should mean those safety devices that control the flow of milk, cream, or frozen dessert mix in relation to the temperature of the product, or heating medium and/or pressure, vacuum or other auxiliary equipment. Flow controls should not be considered as part of the temperature control equipment. Flow controls should be of the flow-diversion type, which automatically cause the diversion of the product in response to a sublegal pasteurization temperature. At sublegal temperatures, flow-diversion devices return the product to the raw-product side of the heating system continuously until legal pasteurization temperatures are obtained, at which time the device restores forward flow through the pasteurizer.

Flow Diversion Device Criteria—All flow diversion devices used in continuous flow pasteurizers should comply with the following or equally satisfactory specifications:

- 1. Forward flow of subtemperature product should be prevented by making all flow promoting devices shut down when the product is below the pasteurization temperature and the valve is not in the fully diverted position or by other equally satisfactory means.
- 2. When a packing gland is used to prevent leakage around the actuating stem, it should be impossible to tighten the stem packing nut to such an extent as to prevent the valve from assuming the fully diverted position.
- 3. A leak escape should be installed on the forward flow side of the valve seat. The leak escape should lie between two valve seats or between two portions of the same seat, one upstream and the other downstream from the leak escape. The leak escape should be designed and installed to discharge all leakage to the outside, or to the constant-level tank through a line separate from the diversion line, provided that when leakage is discharged to the constant-level tank, a sight glass should be installed in a vertical portion of the leak escape line to provide a visual means of leak detection.
- 4. The closure of the forward flow seat should be sufficiently tight so that leakage past it will not exceed the capacity of the leak escape divide, as evidenced when the forward flow line is disconnected; and, in order that proper seating may not be disturbed, the length of the connecting rod should not be adjustable by the user.
- 5. The flow diversion device should be so designed and installed that failure of the primary motivating power should automatically divert the flow of product.
- 6. The flow diversion device should be located downstream from the holder. The flow control sensor should be located in the product line not more than 18 inches upstream from the flow control device.
- 7. In the case of higher heat, shorter time (HHST) pasteurizing systems utilizing the temperatures of 191°F and above and holding times of one second and less, the flow diversion device may be located downstream from the regenerator and/or cooler section, provided that when the flow diversion device is located downstream from the regenerator and/or cooler section, the flow diversion device should be automatically prevented from

assuming the forward flow position until all product contact surfaces between the holding tube and the flow diversion device have been held at or above the required pasteurization temperature continuously and simultaneously for at least the required pasteurization time.

- 8. The pipeline from the diversion port of the flow diversion device should be self-draining and should be free of restrictions or valves, unless such restrictions or valves are constructed to be noticeable and are so designed that stoppage of the diversion line cannot occur.
- 9. When it is used, the pipeline from the leak detector port of the flow diversion device should be self-draining and should be free of restrictions or valves.

#### Flow Controller Instrumentation

The following requirements should be met with respect to the instrumentation of the flow controller:

- 1. The thermal limit controller should be set and sealed so that forward flow of product cannot start unless the temperature at the controller sensor is above the required pasteurization temperature nor continue when the temperature is below that which is required. The seal should be applied by the regulatory agency after testing and should not be removed without immediately notifying the regulatory agency. The system should be so designed that no product can be bypassed around the controller sensor, which should not be removed from its proper position during the pasteurization process. The cutin and cutout temperatures, as shown by the indicating thermometer, should be determined at the beginning of each day's operation and entered upon the recorder chart daily by the plant operator.
- 2. In the case of HHST pasteurization systems, utilizing the temperature of 191°F and above, and holding times of one second or less, with the flow diversion device located downstream from the regenerator and/or cooler section, additional temperature controllers and timers should be interwired with the thermal limit controller, and the control system should be set and sealed so that forward flow of product cannot start until all product contact surfaces between the holding tube and the flow diversion device have been held at or above the required pasteurization temperature, continuously and simultaneously for at least the required pasteurization time. The control system should also be set and sealed so that forward flow cannot continue when the temperature of the product in the holding tube is below the required pasteurization temperature. The seal should be applied by the regulatory agency after test and should not be removed without immediately notifying the regulatory agency. The system should be so designed that no product can be bypassed around the control sensors, which should not be removed from their proper position during the pasteurization process. For these HHST systems, daily measurement by the operator of the cutin and cutout temperatures is not required.
- 3. Manual switches for the control of pumps, homogenizers, or other devices that produce flow through the holder, should be wired so that the circuit is completed only when the product is above the required pasteurization temperature, or when the diversion device is in the fully diverted position.

# Holding Tube

1. Holders should be designed to provide for the holding of product for at least the time required.

- 2. The holder should be so designed that the simultaneous temperature difference between the hottest and coldest product in any cross section of flow at any time during the holding period will not be greater than 1°F. This requirement may be assumed to have been satisfied without test in tubular holders of seven inches or smaller diameter that are free of any fittings through which product may not be thoroughly swept.
- 3. No device should be permitted for short-circuiting a portion of the holder to compensate for changes in rate of flow. Holding tubes should be installed so that sections of pipe cannot be left out, resulting in a shortened holding time.
- 4. The holding tube should be arranged to have a continuously upward slope in the direction of flow of not less than 0.25 inch per foot.
- 5. Supports for tubes should be provided to maintain all parts of holding tubes in a fixed position, free from any lateral or vertical movement.
- 6. The holder should be so designed that no portion between the inlet and the flow control temperature sensor is heated.
- 7. The holding time for the HHST processes should be determined from the pumping rate rather than by the salt conductivity test because of the short holding tube. The holding tube length should be such that the fastest flowing particle of any product will not traverse the holding tube in less than the required holding time. Since laminar flow (the fastest flowing particle travels twice as fast as the average flowing particle) can occur in the holding tube during pasteurization of high-viscosity products, holding-tube lengths are calculated as twice the length required to hold the average flow for the time standard.
- 8. With steam-injection processes, the holding time is reduced because the product volume increases as the steam condenses to water during heating in the injector. This surplus water is evaporated as the pasteurized product is cooled in the vacuum chamber. For example, with a 120°F increase by steam injection, which is probably the maximum temperature rise that will be used, a volume increase of 12% will occur in the holding tube. The measurement of the average flow rate at the discharge of the pasteurizer does not reflect this volume increase in the holding tube. However, this volume increase, i.e., holding time decrease, should be considered in the calculations.
- 9. With a steam-injection process, a pressure-limit indicator is needed in the holding tube to keep the heated product in the liquid phase. The instrument should have a pressure switch so that the flow diversion device will move to the divert position if the product pressure falls below a prescribed value. For operating temperatures between 191°F and 212°F the pressure switch should be set at 10 pounds per square inch (psi). For units that have operating temperatures above 212°F, the pressure switch should be set at a pressure 10 psi above the boiling pressure of the product at its maximum temperature in the holding tube.
- 10. With a steam-injection process, a differential pressure limit indicator across the injector is needed to ensure adequate isolation of the injection chamber. The instrument should have a differential pressure switch so that the flow diversion device will move to the divert position if the pressure drop across the injector falls below 10 psi.
- 11. The process should be as free as possible of noncondensable gases that may evolve from the product or be carried in the steam supply. Any two-phase flow caused by the noncondensable gases would displace the product in the holding tube, resulting in reduced residence times. In addition, these gases in the steam supply may also markedly alter the condensation mechanism at the point of injection. Accordingly, the steam boiler should be supplied with a deaerator. The deaerator will aid in keeping the product in the holding tube as free as possible of noncondensable gases.

# Indicating and Recording Thermometers

- 1. An indicating thermometer should be located as near as practicable to the temperature sensor of the recorder/controller but may be located a short distance upstream from the latter where product between the two thermometers does not differ significantly in temperature.
- 2. The temperature shown by the recorder/controller should be checked daily by the plant operator against the temperature shown by the indicating thermometer. Readings should be recorded on the chart. The recorder/controller should be adjusted to read no higher than the indicating thermometer.
- 3. The recorder/controller charts should be retained for as long as product shelf life plus an appropriate interval. The use of such charts should not exceed the time limit for which they are designed. Recorded data may not overlap. The following information should be entered on the charts as applicable:

#### Date

Number or location of recorder when more than one is used

Extent of holding period

Reading of indicating thermometer at a given time or reference point as indicated on the chart

A record of the time during which the flow diversion device is in the forward flow position

The cutin and cutout milk temperatures recorded daily by the operator at the beginning of the run

Amount and name of pasteurized milk or milk product represented by each batch or run on the chart

Record of unusual occurrences Signature or initials of operator Name of milk plant

## Flow Promoting Devices

- 1. The pump or pumps and other equipment that may produce flow through the holder should be located upstream from the holder, provided that pumps and other flow promoting devices may be located downstream from the holder if means are provided to eliminate negative pressure between the holder and the inlet to such equipment. When vacuum equipment is located downstream from the holder, an effective vacuum breaker, plus an automatic means of preventing a negative pressure in the line between the flow diversion device and the vacuum chamber, should be acceptable.
- 2. The speed of pumps or other flow promoting devices governing the rate of flow through the holder should be so controlled as to insure the holding of every particle of product for at least the time required. In all cases, the motor should be connected to the metering pump by means of a common drive shaft, or by means of gears, pulleys, or a variable-speed drive with the gear box, the pulley box, or the setting of the variable speed protected in such a manner that the holding time cannot be shortened without detection by the regulatory agency. This should be accomplished by the application of a suitable seal(s) after tests by the regulatory agency and such seal should not be broken without immediately notifying the regulatory agency. The provision should apply to all homogenizers used as timing pumps. Variable-speed drives used in connection with the metering pump should be so constructed that wearing or stretching of the belt results in a

slowdown, rather than a speedup, of the pump. The metering or timing pump should be of the positive displacement type or should comply with current FDA specifications for magnetic flow meter systems. Timing pumps and homogenizers, when used as timing pumps, should not have bypass lines connected from their outlet pipelines to their inlet pipelines during processing if an additional flow promoting or vacuum producing device is located within the system. When a homogenizer is used in conjunction with a timing pump, it should be either

- a. Of larger capacity than the timing pump, in which case an unrestricted, open recirculation line should be used to connect the outlet pipeline from the homogenizer to its inlet line. The recirculation line should be of at least the same or larger diameter than the inlet pipeline feeding product to the homogenizer. A check valve, allowing flow from the outlet line to the inlet line, may be used in the recirculating line provided it is of the type that provides a cross-sectional area at least as large as the recirculating line.
- b. Of smaller capacity than the timing pump, in which case a relief line and valve should be used. Such relief line should be located after the timing pump and before the inlet to the homogenizer and should return product to the balance tank or to the outlet of the balance tank upstream of any booster pump or other flow promoting device.
- c. For those systems that do not homogenize all products and wish to utilize a bypass line to bypass the homogenizer while processing such product, the bypass line should be connected with valves that are so designed that both lines cannot be open at the same time. This may be accomplished with three-way plug valves with properly designed and operating pins or other automatic, fail-safe valves that accomplish the same objective.
- 3. The holding time should mean the flow time of the fastest particle of product, at or above the required pasteurization temperature throughout the holder section, i.e., that portion of the system that is outside the influence of the heating medium and slopes continuously upward in the downstream direction, and is located upstream from the flow diversion device. Tests for holding time should be made when all equipment and devices are operated and adjusted to provide for maximum flow. When a homogenizer is located upstream from the holder, the holding time should be determined with the homogenizer in operation with no pressure on the homogenizer valves. For those systems that do not homogenize all product and utilize bypass lines as outlined above, the holding time should be tested in both flow patterns and the fastest time used. The holding time should be tested during both forward and diverted flow. If it is necessary to lengthen the holding time during diverted flow, an identifiable restriction may be placed in the vertical portion of the diversion pipeline. When vacuum equipment is located downstream from the holder, the holding time should be tested with the metering pump operating at maximum flow and the vacuum equipment adjusted to provide for the maximum vacuum. The holding time should be tested in both forward and diverted flow by the regulatory agency initially; semiannually thereafter; after any alteration or replacement that may affect the holding time; and whenever the seal of the speed setting has been broken.

Magnetic Flow Meter-Based Timing Systems

A magnetic flow meter and a meter-based timing system (MBTS) may be used as a replacement for a positive timing pump if the criteria in this section are met.

These systems are of two basic types:

Those employing a constant speed centrifugal pump and a control valve, or those employing an AC variable frequency motor speed control for the centrifugal pump.

1. COMPONENTS—Magnetic flow meter-based timing systems should consist of the following components:

- a. A sanitary magnetic flow meter that has been reviewed by USPHS/FDA or one that is equally accurate and reliable and will produce six consecutive measurements of holding time within one-half (0.5) second of each other.
- b. Suitable converters for conversion of electric and/or air signals to the proper mode for the operation of the system.
- c. A suitable flow recorder capable of recording flow at the flow alarm set point and also at least five gallons per minute higher than the flow alarm setting. The flow recorder should have an event pen, which should indicate the position of the flow alarm with respect to the flow rate.
- d. A flow alarm with an adjustable set point should be installed within the system that will automatically cause the flow diversion device to be moved to the divert position whenever excessive flow rate causes the product holding time to be less than the legal holding time for the pasteurization process being used. The flow alarm should be tested by the regulatory agency at the frequency specified, and the alarm adjustment should be sealed.
- e. A loss-of-signal alarm should be installed with the system that will automatically cause the flow diversion device to be moved to the divert position whenever there is a loss of signal from the meter. The loss-of-signal provision should be tested by the regulatory agency at the frequency specified and should be sealed.
- f. When the legal flow rate has been reestablished following an excessive flow rate, a time delay should be instituted that will prevent the flow diversion device from assuming the forward flow position until at least a 15 second (milk) or 25 second (frozen dessert mix) continuous legal flow has been reestablished. The time delay should be tested by the regulatory agency and if it is of the adjustable type should be sealed.
- g. When a constant speed centrifugal pump is used, a sanitary spring-loaded-to-close, air-to-open control valve should be used to control the rate of flow of product through the HTST system.
- h. When an AC variable-frequency motor speed control is used on the centrifugal timing pump, the control valve is not needed, as the flow rate of product through the system is controlled by feeding the signal from the magnetic flow meter to a controller, which in turn varies the AC frequency to the pump motor, thus controlling the flow rate of product through the system. With these AC variable frequency systems, a sanitary product check valve is needed in the sanitary milk pipe line to prevent a positive pressure on the raw milk side of the regenerator whenever a power failure, shutdown, or flow diversion occurs.
- i. When a regenerator is used with large systems, it will be necessary to bypass the regenerator during startup and when the flow diversion device is in the diverted flow position. Care should be taken in the design of such bypass systems to assure that a dead end does not exist. A dead end could allow product to remain at ambient temperature for long periods of time and allow bacterial growth in the product. Caution should also be observed with such bypass systems and any valves used in them so that raw milk product will not be trapped under pressure in the raw regenerator plates and not have free drainage back to the constant level tank when shutdown occurs.
- j. Most systems will utilize a dual stem flow diversion device and will be using the centrifugal pump during the CIP cleaning cycle. All public health controls required of such systems should be applicable. When switching to the CIP position, the flow diversion device should move to the divert position and should remain in the diverted flow position for at least 10 minutes, regardless of temperature, and the booster pump cannot run during this 10-minute time delay.

- k. All systems should be designed, installed, and operated so that all applicable tests in Appendix I of the current edition of the Grade A Pasteurized Milk Ordinance can be performed by the regulatory agency at the frequency specified. Where adjustment or changes can be made to these devices or controls, appropriate seals should be applied after testing so that changes cannot be made without detection.
- l. Except for those requirements directly related to the physical presence of the metering pump, other requirements of the Grade A Pasteurized Milk Ordinance may be applicable.
- 2. PLACEMENT OF COMPONENTS—Individual components in the magnetic flow meter based timing systems should comply with the following placement condition:
- a. The centrifugal pump should be located downstream from the raw milk regenerator section if a regenerator is used.
- b. The magnetic flow meter should be placed downstream from the centrifugal pump. There should be *no* intervening flow promoting components between the centrifugal pump and the meter, or between the meter and the flow diversion device.
- c. The control valve used with the constant speed centrifugal pump should be located downstream of the magnetic flow meter.
- d. The centrifugal pump, the magnetic flow meter, the control valve, when used with the constant speed centrifugal pump system and the sanitary product check valve, and when used with the AC variable frequency motor speed control system should all be located upstream from the start of the holding tube.
- e. All flow promoting devices that are upstream of the flow diversion device, such as centrifugal timing pumps (constant speed or AC variable frequency motor control types), booster pumps, stuffer pumps, separators, and clarifiers should be properly interwired with the flow diversion device so that they may run and produce flow through the system at sublegal temperatures, only when the flow diversion device is in the fully diverted position when in product run mode. Separators or clarifiers that continue to run after power to them is shut off should be automatically valved out of the system with fail-safe valves so that they are incapable of producing flow.
- f. There should be no product entering or leaving the system (i.e., cream or skim from a separator or other product components) between the centrifugal pump and the flow diversion device.
- g. The magnetic flow meter should be so installed that the product has contact with both electrodes at all times when there is flow through the system. This is most easily accomplished by mounting the flow tube of the magnetic flow meter in a vertical position with the direction of flow from the bottom to the top. However, horizontal mounting is acceptable when other precautions are taken to assure that both electrodes are in contact with product. They should not be mounted on a high horizontal line, which may be only partially full and thereby trap air.
- h. The magnetic flow meter should be piped in such a manner that at least 10 pipe diameters of straight pipe exists upstream and downstream from the center of the meter before any elbow or change of direction takes place.

## Prevention of Product Adulteration with Added Water

1. When culinary steam is introduced directly into the mix downstream from the flow diversion device, means should be provided to preclude the addition of steam to the product, unless the flow diversion device is in the forward flow position. This

provision may be satisfied by the use of an automatic steam control valve with temperature sensor located downstream from the steam inlet, or by the use of an automatic solenoid valve installed in the steam line and so wired through the flow diversion device controls that steam cannot flow unless the flow diversion device is in the forward flow position.

- 2. When culinary steam is introduced directly into the product, automatic means should be provided to maintain a proper temperature differential between incoming and outgoing mix to preclude dilution with water.
- 3. Where a water feed line is connected to a vacuum condenser, and the vacuum condenser is not separated from the vacuum chamber by a physical barrier, means should be provided to preclude the backup and overflow of water from the vacuum condenser to the vacuum chamber. This provision may be satisfied by the use of a safety shutoff valve, located on the water feed line to the vacuum condenser, automatically actuated by a control will shut off the in-flowing water if, for example, the condensate pump stops and the water level rises above a predetermined point in the vacuum condenser. This valve may be actuated by water, air, or electricity and should be so designed that failure of the primary motivating power will automatically stop the flow of water into the vacuum condenser.

## Regenerative Heating (Product to Product)

To prevent contamination of the pasteurized product in regenerators, the raw product should always be under less pressure than the pasteurized product or the heat transfer medium. In the case of product-to-product regenerators, this requirement is necessary to prevent contamination of the pasteurized product with raw product if flaws should develop in the metal or in the joints separating the two. Pasteurizers and aseptic processing systems employing product-to-product regenerative heating with both sides closed to the atmosphere need to meet the following or equally satisfactory specifications:

- 1. Regenerators should be constructed, installed, and operated so that pasteurized or aseptic product in the regenerator will automatically be under greater pressure than raw product in the regenerator at all times.
- 2. The pasteurized or aseptic product, between its outlet from the regenerator and the nearest point downstream open to the atmosphere, should rise to a vertical elevation of 12 inches above the highest raw-product level downstream from the constant-level tank, and should be open to the atmosphere at this or a higher elevation.
- 3. The overflow of the top rim of the constant-level raw tank should always be lower than the lowest product level in the regenerator.
- 4. No pump or flow promoting device that can affect the proper pressure relationships within the regenerator should be located between the pasteurized or aseptic product outlet from the regenerator and the nearest downstream point open to the atmosphere.
- 5. No pump should be located between the raw inlet to the regenerator and the raw supply tank, unless it is designed and installed to operate only when product is flowing through the pasteurized or aseptic product side of the regenerator, and when the pressure of the pasteurized or aseptic product is higher than the maximum pressure produced by the pump. This may be accomplished by wiring the booster pump so that it cannot operate unless
  - a. The metering pump is in operation.
  - b. The flow diversion device is in forward flow position.

- c. The pasteurized or aseptic product pressure exceeds, by at least one psi, the maximum pressure developed by the booster pump. Pressure gauges should be installed at the raw inlet to the regenerator and the pasteurized or aseptic product outlet of the regenerator or the outlet of the cooler. The accuracy of required pressure gauges should be checked by the regulatory agency on installation, quarterly thereafter, and following repair or adjustment.
- 6. The motor, casing, and impeller of the booster pump should be identified, and such records thereof maintained as directed by the regulatory agency. All electric wiring interconnections should be in permanent conduit (except that rubber-covered cable may be used for final connections), with no electrical connections to defeat the purpose of any provisions of this guideline.
- 7. All raw product in the regenerator will drain freely back into the constant-level raw tank when the raw pump(s) are shut down and the raw outlet from the regenerator is disconnected.
- 8. When separators or vacuum equipment are located downstream from the flow diversion device, means should be provided to prevent the lowering of the pasteurized or aseptic product level in the regenerator during periods of diverted flow or shutdown. An effective vacuum breaker, plus an automatic means of preventing a negative pressure, should be installed in the line between the vacuum chamber and the pasteurized or aseptic product inlet to the regenerator.
- 9. In the case of HHST pasteurization systems utilizing the temperature of 191°F and above and holding times of one second or less, with the flow diversion device located downstream from the regenerator and/or cooler section, the requirement that the outlet of the regenerator or cooler should rise to a vertical elevation of 12 inches above the highest raw-product level downstream from the constant-level tank and should be open to the atmosphere at this or a higher elevation, may be eliminated, provided that a differential pressure controller is used to monitor the highest pressure in the raw-product side of the regenerator and the lowest pressure in the pasteurized side of the regenerator, and the controller is interlocked with the flow diversion device and is set and sealed so that whenever improper pressures occur in the regenerator, forward flow of product is automatically prevented and will not start again until all product contact surfaces between the holding tube and the flow diversion device has been held at or above the required pasteurization temperature, continuously and simultaneously for at least the required pasteurization time.

In the case of aseptic processing systems used for producing aseptic products, there should be an accurate differential pressure recorder-controller installed on the regenerator. Each inch working scale on the chart may not display more than 20 pounds per square inch. The chart scale divisions may not exceed two pounds per square inch. The controller should be tested for accuracy against a known accurate standard pressure indicator upon installation and at least once every three months of operation thereafter or more frequently if necessary to ensure its accuracy. One pressure sensor should be installed at the aseptic product regenerator outlet, and the other pressure sensor should be installed at the raw-product regenerator inlet.

10. When culinary steam is introduced directly into products, as the means of terminal heating to achieve pasteurization or aseptic processing temperature, and vacuum equipment is located downstream from the holding tube, the requirement that a vacuum breaker be installed at the inlet to the pasteurized or aseptic side of the regenerator may be eliminated, provided that the differential pressure controller is installed and wired to control the flow diversion device as described in paragraph 9 of this section.

11. When the differential pressure controller is installed and wired to control the flow diversion device as described in paragraph 9 of this section, the raw-product booster pump may be permitted to run at all times, provided that the metering pump is in operation.

## Regenerative (Product-to-Water-to-Product) Heating

Product-to-water-to-product regenerators with both the product and the heat-transfer water in the raw section closed to the atmosphere should comply with the following or equally satisfactory specifications:

- 1. Regenerators of this type should be so designed, installed, and operated that the heat transfer medium side of the regenerator in the raw-product section will automatically be under greater pressure than the raw-product side at all times.
- 2. The heat transfer water should be safe water, and the heat transfer water should be in a covered tank that is open to the atmosphere at an elevation higher by at least 12 inches than any raw product level downstream from the constant-level tank. The heat transfer water between its outlet from the regenerator and the nearest point downstream open to the atmosphere should rise to a vertical elevation of at least 12 inches above any raw product in the system and should be open to the atmosphere at this or a higher elevation.
- 3. The heat transfer water circuit should be full of water at the beginning of the run, and all loss of water from the circuit should be automatically and immediately replenished whenever raw product is present in the regenerator.
- 4. The overflow of the top rim of the constant level raw tank should always be lower than the lowest product level in the raw section of the regenerator. The regenerator should be designed and installed so that all raw product should drain freely back to the upstream supply tank when the raw-product pumps are shut down and the raw-product line is disconnected from the regenerator outlet.
- 5. No pump should be located between the raw inlet to the regenerator and the raw-product supply tank unless it is designed and installed to operate only when water is flowing through the heat transfer section of the regenerator, and when the pressure of the heat transfer water is higher than the pressure of the raw product. This may be accomplished by wiring the booster pump so that it cannot operate unless
  - a. The heat transfer water pump is in operation.
- b. The heat transfer water pressure exceeds by at least one pound per square inch the raw product pressure in the regenerator. Pressure gauges should be installed at the raw product inlet and the heat transfer water outlet of the regenerator. The accuracy of the required pressure gauges should be checked by the regulatory agency on installation and quarterly thereafter and following repair or replacement.
- 6. Product-to-water-to-product regenerators alternatively may be constructed, installed, and operated so that the product in the regenerator will be under greater pressure than the heat transfer medium in the aseptic product side of the regenerator.
- a. The differential pressure recorder-controller should be used to monitor pressures of the aseptic product and the heat transfer medium. One pressure sensor should be installed at the aseptic product outlet of the regenerator and the other pressure sensor should be installed at the heat transfer medium inlet of the aseptic product side of the regenerator. This recorder-controller should divert the flow diversion device whenever the lowest pressure of the aseptic product side of the regenerator does not exceed the heat

transfer medium pressure by at least 1 psi. Forward flow of product should be automatically prevented until all product-contact surfaces between the holding tube and the flow diversion device have been held at or above the required sterilization temperature continuously and simultaneously for at least the sterilization time.

b. The heat transfer medium pump should be wired so that it cannot operate unless the metering pump is in operation.

## Indicating Thermometer Located on Pasteurization Pipelines

Type—Mercury-actuated, direct-reading, contained in corrosion-resistant case that protects against breakage and permits easy observation of column and scale, filling above mercury, nitrogen, or equally suitable gas. Provided that types other than mercury-actuated may be used when they have been (1) demonstrated to be equally fail-safe, accurate, reliable, and meet the scale and thermometric response specifications and (2) approved by the regulatory agency.

Magnification of mercury column—To apparent width of not less than 0.0625 of an inch.

Scale—Should have a span of not less than 25°F including the pasteurization temperature plus or minus 5°F; graduated in 0.5°F divisions with not more than 8°F per inch of scale; protected against damage at 220°F and in the case of thermometers used on HHST systems, protected against damage at 300°F.

Accuracy—Within 0.5°F plus or minus throughout specified scale span.

Stem Fittings—Pressure-tight seat against inside wall of fittings; no threads exposed to milk; distance from underside of ferrule to top of the sensitive portion of bulb not less than three inches.

Thermometric Response—When the thermometer is at room temperature and then is immersed in a well-stirred water bath 19°F or less above the pasteurization temperature, the time required for the reading to increase from water-bath temperature minus 19°F to water-bath temperature minus 7°F should not exceed 4 seconds.

Bulb—Corning normal, or equally suitable thermometric glass.

## Recorder/Controllers For Continuous Pasteurizers

Case—Moisture proof under normal operating conditions in pasteurization plants.

Chart Scale—Should have a span of not less than 30°F, including the temperature at which diversion is set, plus or minus 12°F, graduated in temperature scale divisions of 1°F spaced not less that 0.0625 of an inch apart at the diversion temperature, plus or minus 1°F, provided that temperature-scale divisions of 1°F spaced not less than 0.040 of an inch apart are permitted when the ink line is thin enough to be easily distinguished from the printed line, graduated in time-scale divisions of not more than 15 minutes, having an equivalent 15-minute cord or straight-line length of not less than 0.25 of an inch at the diversion temperature, plus or minus 1°F.

Temperature Accuracy—Within 1°F, plus or minus, at the temperature at which the controller should be electrically operated.

Power Operated—All recorder/controllers for continuous pasteurization should be electrically operated.

Pen Arm Device—Easily accessible; simple to adjust.

Pen and Chart Paper—Pen designed to give line not over 0.025 of an inch wide; easy to maintain.

Temperature-sensing Device—(Bulb, tube, spring, thermistor) protected against damage at temperature of 220°F, provided that recorder/controller temperature sensing devices used on HHST systems should be protected against damage at temperatures of 300°F.

Submerged Stem Fitting—Pressure-tight seat against inside wall of pipe, no threads exposed to milk or milk products, location from underside of ferrule to the sensitive portion of the bulb not less than 3 inches.

Chart Speed—A circular chart should make one revolution in not more than 12 hours. Two charts should be used if operations extend beyond 12 hours in one day. Circular charts should be graduated for a maximum record of 12 hours. Strip charts may show a continuous recording over a 24-hour period.

Frequency Pen—The recorder/controller should be provided with an additional pen arm for recording, on the outer edge of the chart, the record of the time at which the flow control device is in the forward flow, diverted flow, or stopped position. The chart time line should correspond with the reference arc, and the recording pen should rest upon the time line matching the reference arc.

Controller—Actuated by the same sensor as the recorder pen but cutin and cutout response independent of pen arm movement.

Controller Adjustment—Mechanism for adjustment of response temperature simple, and so designed that the temperature setting cannot be changed or the controller manipulated without detection.

Thermometric Response—With the recorder/controller bulb at room temperature and then immersed in a well-stirred water or oil bath at 7°F above the cutin point, the interval between the moment when the recording thermometer reads 12°F below the cutin should be not more than five seconds.

Chart Support Drive—The rotating chart support drive should be provided with a pin to puncture the chart in a manner to prevent its fraudulent rotation.

*Note*: In the case of recorder controllers with a high and low setting for diversion temperatures, the chart should indicate whether the recorder is at the high or low setting. This can be accomplished by the operator doing a cutin and cutout test each time the setting is changed.

## Equipment Tests and Examination

The state or local regulatory agency should perform the indicated tests on the following instruments and devices initially on installation, and at least once each three months thereafter, and whenever any alteration or replacement is made may affect the proper operation of the instrument or device, provided that the holding time test should be conducted at least every six months.

Instrument or device	Test number <sup>a</sup>	Test objective
HTST and Aseptic Processing (AP) indicating thermometer	1	Accuracy
HTST and AP indicating thermometer	7	Thermometric response
HTST and AP recording thermometer	2	Temperature accuracy
HTST and AP recording thermometer	3	Time accuracy
HTST and AP recorder controller	2	Temperature accuracy

Instrument or device	Test number <sup>a</sup>	Test objective
HTST and AP recorder controller	4	Check reading of recorder controller against indicating thermometer
HTST and AP recorder controller	8	Thermometric response
HTST and AP recorder controller	10	Confirm cutin and cutout temperatures
HTST and AP flow diversion device	5	Assembly and function
HTST and AP auxiliary (booster) pump	9	Function of automatic control devices
HTST and AP auxiliary (booster) pump	9	Accuracy of pressure gauges' holding time
HTST and AP system	12	Thermal limit control for sequence logic
HTST and AP system	13	Setting of control switches for product pressure in the holding tube
HTST and AP system	14	Setting of control switches for differential pressure across the injector

<sup>&</sup>lt;sup>a</sup> The test numbers refer to test procedures accepted by the FDA and listed in Appendix H of the current edition of the Grade "A" Pasteurized Milk Ordinance.

# **Drying of Frozen Dessert Mix**

#### Risk L to H

Frozen dessert mix to be dried should be produced in accordance with the provisions of these guidelines. Additional guidance as to condensing and drying equipment and practices can be found in

- 1. The latest edition of the *Grade A Condensed and Dry Milk Products and Condensed and Dry Whey—Supplement to the Grade A Pasteurized Milk Ordinance*.
- 2. The current applicable 3A standards and practices. These sources should be used to determine the risk factors involved in conditions observed at a frozen dessert mix condenser or dryer.

## **Bulk Tank Transported Pasteurized Mix**

The FDA's dairy initiatives have shown that problems may occur when frozen dessert mix and other dairy ingredients are pasteurized at one location and transported to another plant for further processing without being repasteurized. This bulk product is more susceptible, since the product is handled and exposed to potential contamination.

Risk H In order to prevent this potential for contamination, frozen dessert mix should be packaged in the plant where it is pasteurized. Mix shipped in bulk tank trucks to another location should be repasteurized at that plant prior to freezing and packaging.

If properly handled and protected, packaged frozen dessert mix in sealed containers can be safely transported from one plant to another for freezing without repasteurization.

Risk H If pasteurized product for repasteurization is loaded in a raw-product receiving area, particular attention should be paid to product and CIP connections so that raw product in lines and tanks is never directly connected to any line which extends back to pasteurized product lines or tanks. A physical break is needed; closed valves are not enough.

Risk M The truck bay from which pasteurized product is loaded out should meet all of the same requirements as a raw tank truck receiving bay.

# **Ingredients Added After Pasteurization**

Risk H All dairy products (milk solids, whey, nonfat dry milk, condensed milk, cream, skim milk, etc.), eggs, egg products, cocoa, cocoa products, emulsifier, stabilizers, liquid sweeteners, and dry sugar should be added prior to pasteurization.

All reconstitution or recombination of dry, powdered, or condensed ingredients with water should be done prior to pasteurization.

The only ingredients that may be added after pasteurization are those flavoring and coloring\* ingredients that are

- 1. Subjected to prior heat treatment sufficient to destroy pathogenic microorganisms
- 2. Of 0.85\* water activity or less
- 3. Of pH less than 4.7
- 4. Roasted nuts (added at the freezer)
- 5. High in alcohol content
- 6. Bacterial cultures
- 7. Fruits and vegetables added at the freezer<sup>†</sup>
- 8. Subjected to any other process that will assure that the ingredient is free of pathogenic microorganisms

## Freezing and Packaging

There is demonstrated risk of postpasteurization contamination during freezing and packaging of frozen desserts. In view of this, virtually all applicable sections of these guidelines take on increased significance when applied to this portion of the plant.

The exposure of frozen desserts and frozen dessert mixes and frozen dessert containers and lids on packaging machines during packaging has been related to pathogen contamination of finished frozen desserts.

Hand-packing can also result in the exposure to contamination, which would nullify the effect of pasteurization.

Package Design

Risk M Packages that are not designed properly to protect product during storage and dispensing can allow similar contamination.

<sup>\*</sup>Some colorants have a history of bacterial contamination. Careful monitoring and extra precautions may be needed to assure finished-product safety.

<sup>&</sup>lt;sup>†</sup> A plant quality assurance program is necessary to assure that the fresh fruit and vegetable products are of high quality and do not contaminate the dairy product.

Caps and closures for mix or other fluid products should be designed so that the pouring lip is adequately protected. To prevent contamination, lids of tub and canister-type containers for frozen desserts should be designed to overlap the tub or container or be overwrapped. Other types of frozen dessert packages should provide a similar level of protection. Tamper-evident packaging is strongly recommended.

## Packaging Mix

Risk H Packaging mix should be done at the pasteurization plant and in acceptable mechanical equipment. Adequate drip deflectors are needed on each filler valve. Conveyor in-feed lines of packaging should have effective overhead shielding from the point the packages are formed or loaded on the machine.

The level of protection and shielding should continue until the container has been sealed. Condensate from the inner portions of the machine should not drip onto mandrels or into packaging. Mandrels which have internal cooling should be adjusted so that they stay relatively dry. Internal mandrel cooling water (with or without glycol) can leak into cartons when a seal fails. The reserve tanks for this cooling media should be adequately protected and should be coliform and pathogen free. Air under pressure directed at product contact surfaces should meet the criteria of this guideline.

If defoamers are used, they should not return product or foam to the filler bowl.

#### Packaging Frozen Desserts

Both bulk frozen desserts (pint-size containers and larger) and novelties will be addressed in this section. Each will include general comments about freezing and packaging. Some of the comments relate to both but are discussed only once.

Risk H Containers for frozen desserts should be filled and sealed mechanically on properly designed, easily cleanable equipment that has shields and drip deflectors to prevent condensate, water, or other contamination from entering containers from the time they are put on the conveyor feed line until filled and sealed. Similar overhead protection is needed for open containers of package and lids. Packaging machines may be manually operated.

With some reasonable precautions, hand-capping may be acceptable if suitable mechanical equipment for the capping or closing of specific containers is not available. Other methods which eliminate all possibility of contamination may be approved by the regulatory agency.

- Risk H Transfer pumps and ripple pumps should be broken down, inspected, and cleaned after each use and sanitized prior to start up.
- Risk H Mix should not be hand-dumped into the flavor tank. When mix is transferred to the tank, a valve and a pump should be used. All flavor tanks should be kept covered except when flavoring is added. The flavor tank should be thoroughly cleaned after each use.
- Risk H Pails used for rework or adding flavors should be cleaned after each use and sanitized prior to reusing.

- Risk H Measuring containers used to add flavors to the mix should be cleaned and sanitized prior to each use. An area dedicated to the storage of these containers should be available. These measuring containers should only be used for flavors.
- Risk H Variegators should be kept clean. Variegators should be designed so that they can be effectively cleaned and sanitized.
- Risk M–H The freezer should be in good repair and properly constructed. The seal around the shaft, which extends through the back of the freezer to the motor, should be evaluated for cleanliness and leaks. If it is leaking, the seal cannot be adequately cleaned.
- Risk M The air supply to the freezer should be properly filtered.

## Novelties

Generally, novelty machines form, freeze, and package items as a complete process. These machines are designed for a specific purpose and in many cases are not easy to clean properly. Most product contact surfaces are designed to be cleaned out but the housing, drive units, etc. present much more of a challenge. If a machine jams, product can get on the container and then seep into the drive unit and the undercarriage. These nonproduct contact surfaces need to be kept clean.

Most novelties are of two types:

- 1. Stick novelties
- 2. Stickless novelties (extruded)

Other frozen novelties, such as molded ice cream, should be given equal levels of public health protection.

The molds of stick novelties are submerged in brine or glycol, and the product is added in a liquid or semiliquid state. The sticks are inserted, the product is extracted, and coatings, nuts, etc. may then be applied before packaging.

- Risk H The brine is normally calcium carbonate. This is a corrosive chemical that can cause burning to mouth tissue if it is mixed with frozen novelties. In most recently manufactured machines, the molds are sealed away from brine; however, all molds can leak and some older machines are not well sealed. To minimize this potential problem, a bright, distinctive food color should be added to the brine so that any leakage will clearly show on the finished product.
- Risk H The molds should be kept clean.
- Risk H Cleaning and sanitizing procedures for the molds should be reviewed. Adequate shields are needed to protect open molds and molds that are sanitized during each cycle of the machine. Other functions needing daily attention include proper breakdown of hopper filler valves, Tygon hose assembly, and mold filler nozzles.
- Risk H Condensate can build up on extractor bars during the defrost and extraction process. Defrost water velocity and temperature should be controlled to minimize this occurrence. These conditions necessitate detailed cleaning and sanitizing of extractor bars.

- Risk H When a stainless steel chute is used to convey product to the wrapper after extraction, the chute should be cleaned at least every hours during the production run. It would be preferable to update equipment so that the product avoids this chute and drops directly into a package wrapper.
- Risk M Certain stick novelty machines with rubber filler nozzles can drip condensate into the molds. The molds can be protected by maintaining proper room dehumidification and condensate deflection. Further, when the product hopper overflows, product can run down the side of the hopper and into the finished product unless a deflector shield is in place.

Stickless Novelty (Extruded)

The product is extruded in soft form through an extruder nozzle.

All equipment contains some or all of the following items:

- 1. Product extruder
- 2. Cookie or plate dispenser
- 3. Nut dispenser
- 4. Enrober (adds coating)
- 5. Wrapper
- 6. Stick inserter
- Risk H Cleaning the plates and associated parts is one of the biggest problems facing the novelty industry. The chain-driven system takes the plates inside the freezing tunnel. Blowers are used in the tunnel to circulate air for quick freezing. Cleaning is often accomplished by running a series of plates out of the tunnel and cleaning them individually or by continuous hand-brushing and sanitizing. Because of the chain-driven system, they are difficult to clean. When cleaning, care should be taken to avoid getting chain lube on the plates.
- Risk H Cup fillers are difficult to clean. Product contact surfaces and other parts of the machine, particularly the underside of the filler, will accumulate product. At the end of each run, these areas should be cleaned and kept free of product residues. The enrober should also be dismantled and cleaned by hand each day.
- Risk H Clean novelty equipment that is brought into the processing area from storage should be cleaned and sanitized prior to processing.
- Risk H Industrial lubricants with high melting points should be avoided because of problem with coliforms associated with these lubricants.
- Risk H Any air blows, particularly on long lines, should have a sanitary check-valve assembly. Check valves should be manually cleaned each day. Plastic hoses on fillers should be removed and cleaned by hand each day. Extruder heads should have a flexible connection to avoid extrusion of product onto plates during freezer startup and changeover.
- Risk H Water used to glaze product to help prevent sticking to the paper wrapper should be pasteurized or treated to lower the pH. In addition, water dips should have a continuous overflow to minimize product accumulation throughout the product run.

- Risk H Sufficient overhead shielding and protection needs to be provided so that product and packaging are adequately protected against dust, splash, condensate, etc.
- Risk M Cardboard brushes or teflon wipers taped to the machine should not be used to keep product off the round table, i.e., to squeegee off the table.

# **Hardening Rooms and Freezers**

Risk L Hardening rooms should be capable of bringing semifrozen mix rapidly to a hard frozen condition. Freezers should keep product hard-frozen. Thermometers should be present so that this can be monitored. Freezers and hardening rooms need to be kept neat, clean, and relatively free of product spillage.

# **Reclaiming Operations**

Risk M-H

Pathogen contamination of finished frozen desserts and dairy plant environments have been associated with incomplete safeguards in the handling of salvaged or reclaimed product.

Product that has been in distribution channels may have been temperature-abused, tampered with, or exposed to chemical or biological contamination. This product cannot be safely reclaimed.

Safe reclaiming of rework from freezer startup and product changeover as well as filler bowl drainage, tank and line rinsing, and product from defoamer systems can be accomplished. However, this requires strict adherence to the following basic public health concepts:

- 1. Reclaim areas and equipment should be constructed, maintained, and protected at least as well as other normal production and processing areas.
  - 2. Only product that has not left the plant premises should be reclaimed.
- 3. All product to be reclaimed except that from defoamers and tank or line rinsing should be maintained below 45°F. Product salvaged from defoamers and tank or line rinsing should be immediately cooled to below 45°F.
- 4. Packages of product to be reclaimed should be clean and free of contamination. Product from leaking or badly damaged containers should not be reclaimed.
- 5. Packaged product should be opened in such a way as to minimize the potential for contaminations; i.e., containers should not be opened by slashing, smashing, or breaking.
- 6. Because woven wire strainers cannot be effectively cleaned, they should not be used to remove bulky ingredients.
- 7. Reclaim dump stations and tanks should be covered or protected except when product in packages is actually being dumped through the opening.

#### Recommended Standards for Frozen Desserts and Frozen Dessert Mix

Risk M	Temperature (mix only)	Below 45°F
Risk H	Antibiotics	Negative <sup>a</sup>
Risk H	Other drug residues	Negative <sup>a</sup>
Risk H	Pesticides, herbicides, other adulterant residue	Negative <sup>a</sup>
Risk H	Detectable residue from cleaners, sanitizers, or other adulterant	None
Risk M	Standard plate count (except cultured products containing viable organisms)	Not over 30,000/mL
Risk H	Coliform <sup>b</sup> Phosphatase	Not over 10/mL Less than 1 microgram per mL by the Schrarer Rapid Method or equivalent

<sup>&</sup>lt;sup>a</sup> Negative should be interpreted to include any result below a federally defined action or working level.

## **GLOSSARY**

*Address*—A numerical label on each input or output of the computer. The computer uses this address when communicating with the input or output.

Aseptic Processing—When used to describe a frozen dessert mix, the product has been pasteurized as defined in this document, subjected to sufficient heat processing, and packaged in a hermetically sealed container, to conform to the applicable requirements of 21 CFR 113 and maintain commercial sterility of the product under normal nonrefrigerated conditions.

Dairy Ingredient—Any milk derived ingredient.

**Dairy Product**—Means any product that contains any milk derived ingredients.

Fail Safe—Design considerations that cause the instrument or system to move to the safe position upon failure of electricity, air, or other support systems.

*Field Alterable*—A device having a specific design or function that is readily changed by user and/or maintenance personnel.

**Frozen Dessert**—Includes any ice cream, frozen custard, goat's milk ice cream, ice milk, goat's milk ice milk, mellorine, sherbet, or water ice, as well as frozen novelty items made from any of the above. It also includes similar nonstandardized foods such as shake mixes and frozen yogurt products.

Frozen Dessert Mix—Any frozen dessert prior to freezing but after the addition of all ingredients (except those added during the freezing process such as nuts or bulky fruits).

*Frozen Dessert Novelty*—Frozen dessert novelty is a product consisting of a frozen dessert but may include other foods. It is normally produced in single-serving-size units.

Goat's Milk Ice Cream—The food defined in the Code of Federal Regulations, Title 21, Section 135.115.

<sup>&</sup>lt;sup>b</sup> If a positive phosphatase is found, an investigation should be made to determine the cause. If the cause is improper pasteurization, the risk is H and product involved handled accordingly. If the positive comes from other causes, no risk factor should be assigned.

Goat's Milk Ice Milk—The food defined in the Code of Federal Regulations, Title 21, Section 135.125.

*HACCP*—The essential features of a HACCP system include:

- 1. **Hazard Analysis**—The identification and assessment of hazards relating to such areas as the production, processing/manufacturing, storage, and distribution of raw materials, ingredients, and foods. Included in a hazards analysis are the relevant human factors that may affect product safety.
- 2. **Critical Control Points**—The identification of those points in the process where loss or inadequate control over the identified hazards would result in an unacceptable food safety risk.
- 3. **Monitoring**—The establishment of procedures or programs that continuously monitor the critical control points of a process. This monitoring may include physical, chemical, and microbiological testing as well as inspections.

*Ice Cream and Frozen Custard*—The foods defined in the *Code of Federal Regulations*, Title 21, Section 135.110.

Ice Milk—The food defined in the Code of Federal Regulations, Title 21, Section 135.120.

**Plant**—Any place that produces or processes a frozen dessert as defined within the scope of these guidelines.

**Properly Designed and Operated Pasteurization Equipment**—Equipment that is designed, installed, operated, and tested in substantial compliance to the latest edition of the *PMO Grade A Pasteurized Milk Ordinance* with appropriate appendixes and FDA interpretive coded identical memoranda.

Safe-Moisture Level—A level of moisture low enough to prevent the growth of undesirable microorganisms in the finished product under the intended conditions of manufacturing, storage, and distribution. The maximum safe moisture level for a food is based on its water activity. Water activity will be considered safe for a food if adequate data are available that demonstrate that the food at or below the given water activity will not support the growth of undesirable microorganisms.

**Sanitize**—To treat adequately the food contact surfaces by a process that is effective in destroying vegetative cells of microorganisms of public health significance, and in substantially reducing numbers of other undesirable microorganisms but without adversely affecting the product or its safety for the consumer.

**Sherbet**—Sherbet is the food defined in the *Code of Federal Regulations*, Title 21, Section 135.140.

*Standby Status*—The computer is turned on, running and waiting for instructions to start processing input data. This instruction is usually accomplished by a manually operated switch.

**Status Printing**—Some computers are programmed to interrupt printing of the chart record and print the status of key set points and conditions such as cold milk temperature, holding tube temperature, diversion temperature setting, and chart speed.

*Ultra-Pasteurized*—When used to describe frozen dessert mix, such product shall have been thermally processed at or above 280°F for at least two seconds, so as to produce a product that has an extended shelf life under refrigerated conditions.

Water Activity—A measure of the free moisture in a food and the quotient of the water vapor pressure of the substance divided by the vapor pressure of pure water at the same temperature.

Water Ices—The foods defined in the Code of Federal Regulations, Title 21, Section 135.160.