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K. B. Caldwell

H. D. Goff

D. W. Stanley

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A LOW-TEMPERATURE SCANNING ELECTRON MICROSCOPY STUDY OF ICE CREAM.

II. INFLUENCE OF SELECTED INGREDIENTS AND PROCESSES

K. B. Caldwell¹, H. D. Goff, and D. W. Stanley

Department of Food Science, University of Guelph,
Guelph, Ontario, Canada N1G 2W1

¹Present Address: Ault Foods, London, Ontario, Canada, N6B 1N8

Abstract

The objective of this study was to examine the influence of processing parameters, viz., incorporation of polysaccharide stabilizers, freezing rates, and storage times and temperatures on the microstructure of ice cream. As the freezing rate was reduced, ice crystals and air bubbles increased in size. However, quench-freezing also contributed to poor texture; thus an optimum freezing rate exists for the production of suitably-sized ice crystals and texture in ice cream. Model systems of polysaccharide stabilizer solutions were seen to have a characteristic network structure when quench-frozen which was altered by the addition of sucrose. Stabilized ice cream initially had smaller mean ice crystal diameters than unstabilized samples and also held its structure better during storage at fluctuating temperatures as measured through ice crystal growth and sensory analysis.

Key Words: Ice cream, cryo-scanning electron microscopy, low-temperature scanning electron microscopy, polysaccharides, freeze-concentration, freezing rates, storage stability, ice crystals, stabilizers.

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Direct inquiries to H.D. Goff
Telephone number: 519 824 4120 ext. 3878
Fax number: 519 824 6631

Introduction

In the first paper of this series (Caldwell *et al.*, 1992), we reported on the general microstructure of ice cream as viewed by low-temperature scanning electron microscopy (LT-SEM). The ability to examine ice cream in a natural state through LT-SEM makes it possible to observe the influence that ingredients and processing conditions have on ice crystal size and ice cream microstructure. Polysaccharides are commonly added to food as stabilizing agents because they alter the physical properties through modification of the behaviour of water (Sharma, 1981). It has been stated that stabilizers function in ice cream to increase the viscosity of the mix, improve aeration and body, control meltdown and control ice crystal growth during storage (Cottrell *et al.*, 1979; Arbuckle, 1986; Berger, 1990). The mode of action of stabilizers has been discussed in detail elsewhere (Caldwell *et al.*, submitted to J. Dairy Sci.).

Texture of ice cream is initially achieved in the scraped-surface freezer where nucleation is initiated and air is incorporated. Many tiny ice crystal nuclei are then scraped into the bulk of the mix where crystal growth will occur (Hartel, 1990). Once the product leaves the freezer, no further nucleation occurs (Muhr *et al.*, 1986). Hardening to -18 °C core temperature should be achieved quickly to retain smooth texture and small ice crystals (Arbuckle, 1986). Storage at any constant temperature below -18 °C is acceptable because the amount of unfrozen water will remain constant (Hartel, 1990). Quality losses in ice cream occur during temperature abuse (heat shock) whereby large or agglomerated ice crystals grow at the expense of smaller ones. When ice cream is exposed to temperatures above storage temperature, some of the ice crystals melt and, upon lowering of the temperature, this water recrystallizes. In contrast to crystallization in the freezer, this ice recrystallization is a slow quiescent process and takes place on existing crystals (Larsen, 1990). The number of ice crystals decreases and the individual size increases resulting in losses of textural quality due to organoleptic detection of the crystals (iciness). Thus, the objective of

the present study was to examine the influence of selected processing parameters, viz., incorporation of polysaccharide stabilizers, freezing rates, and storage times and temperatures, on the microstructure and quality of the ice cream.

Materials and Methods

Influence of Polysaccharide Stabilizers

Model systems. Model systems of 20 and 40% sucrose solutions were prepared in the presence of stabilizers (xanthan, locust bean gum, carrageenan, guar gum) varying in concentration from 0 to 0.6%. Stabilizer solutions without sucrose were also prepared. Dry solids (sucrose and stabilizer) were blended, water was warmed to the appropriate temperature for each stabilizer before the solids were slowly added, the solutions were vigorously stirred using air-driven agitators, heated until the solutions reached 80 °C, and held for 10 minutes. Solutions containing polysaccharides were homogenized at 6.8 MPa. All solutions were chilled to 5 °C and held for 24 hours before use. Carbohydrate solutions were viewed in the LT-SEM following the method of Caldwell *et al.* (1992) after either being quenched in liquid nitrogen (-210 °C) or frozen in the batch freezer (Taylor, Rockton, IL).

Ice cream. The ice cream mix utilized throughout this study consisted of 11% milk fat, 11% milk solids-not-fat (msnf), 12% sucrose, 4% 42 DE corn syrup solids (Casco, Inc., Etobicoke, Ont.), 0.23% vanilla, 0.15% locust bean gum (LBG) and 0.02% food-grade carrageenan (Food Specialties, Halton Hills, Ont.). The mix was prepared for continuous processing utilizing fresh cream, skim milk and instantized, low-heat non-fat dry milk as sources of milk solids. The mixes were batch-pasteurized at 74 °C for 15 minutes, homogenized in a 2-stage homogenizer (Cherry-Burrell Superhomo, Chicago, IL) at 17.2 MPa and 3.4 MPa, cooled to 5 °C and aged for 24 hours. A slight variation of the above mix was prepared for batch freezing consisting of higher levels of milk fat (13%), and carrageenan (0.2%). In both cases a control mix was prepared with the above formulas minus the stabilizers. Control and stabilized ice cream mixes were compared by continuously freezing the mix in a Vogt VA-80 freezer (Cherry-Burrell, Chicago, IL) or batch freezing in a Taylor freezer (Taylor, Rockton, IL) followed by quiescent hardening and storage at -25 °C according to the experimental procedure outlined previously (Caldwell *et al.*, 1992). Samples were viewed using the LT-SEM procedure described by Caldwell *et al.* (1992).

Influence of Freezing and Hardening Rates

The influence of freezing and hardening rates on both control and stabilized ice creams were studied by

freezing ice cream under batch and continuous processes and exposing the ice cream to three rates of hardening: quiescently at -25 °C, in a plate freezer at -40 °C, or in liquid nitrogen slush at -210 °C. Samples were viewed at 1 day and 2 weeks of storage at -25 °C using the methodology described previously (Caldwell *et al.*, 1992). Unless specified otherwise, all micrographs were from samples etched for 10 minutes. A Zidas Image Analyzer (Zeiss, Germany) was used to calculate cross-sectional area, maximum diameter, minimum diameter and perimeter of the ice crystals. The maximum diameter was taken to be the length of the longest axis of the crystal, although not all crystals were of the same shape.

Influence of Storage Times and Temperatures

Continuously frozen ice cream with and without polysaccharide stabilizer was sampled at storage times of 0, 3 and 24 weeks. Storage temperatures of the samples were fluctuated between -25 °C and -10 °C to simulate a severe heat shocking process. Ice cream was removed from the -25 °C freezer and placed in a -10 °C cabinet for 8 to 10 hours each day during the storage period prior to returning to the -25 °C freezer. Image analyses of ice crystal sizes was performed as above.

Sensory Analyses

Four ice cream samples were used in a ranking sensory panel. Forty-six untrained panelists ranked the samples in sensory analyses facilities for iciness with 1 being the most icy and 4 being the least icy. All products and utensils were equilibrated to -15 °C and scooped immediately for evaluation. Iciness in ice cream was defined to the panelists as the development of a coarse texture by large ice crystals. The four ice creams ranked were: A, control stored at -25 °C; B, control heat-shocked (as above); C, stabilized stored at -25 °C; and D, stabilized heat-shocked. The ranks of the four ice cream samples for each judge were analyzed according to Larmond (1977).

Statistical Analyses

Sample sizes of approximately 100 ice crystals were obtained from at least 10 fields for each sample, each of the hardening conditions (quiescent, plate, and liquid nitrogen quench), and each storage time (0, 3 and 24 weeks). A GLM procedure was used for both experiments (SAS, 1982). The freezing rate experiment had 3 treatments and the storage study had 2 treatments with 3 levels of time each. Both studies were conducted in duplicate. Significance was determined at a level of 5%.

Results and Discussion

Influence of Polysaccharide Stabilizers

Model systems. The influence of freezing rate on

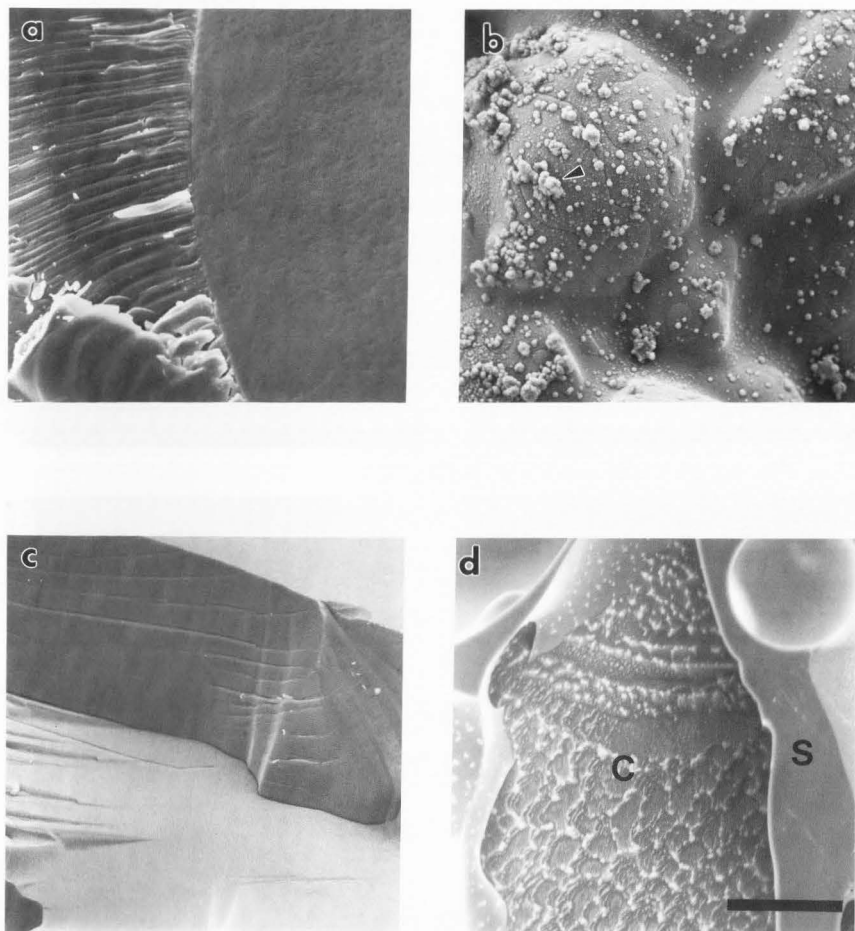


Figure 1. Liquid nitrogen quenching and batch scraped-surface freezing. a) 20% sucrose, quenched; b) 20% sucrose, batch-frozen; c) 40% sucrose, quenched; and d) 40% sucrose, batch-frozen, showing ice crystal socket (C) and serum phase (S). Arrow in Fig. 1b indicates frost contamination. Bar = 25 μm (all four figures are at same magnification).

model sucrose and polysaccharide solutions was investigated by comparing quenching in liquid nitrogen with freezing in a batch freezer. The freezing rate of carbohydrate solutions greatly influence the resultant microstructure. Model solutions of 20 and 40% sucrose as-

sumed a structure resembling solid ice when quench-frozen (Figs. 1a and c). Under slower freezing conditions in the batch freezer, the 20% sucrose solution formed large clumps, with no differentiation of ice or solid phases, whereas the 40% sucrose solution exhibited a

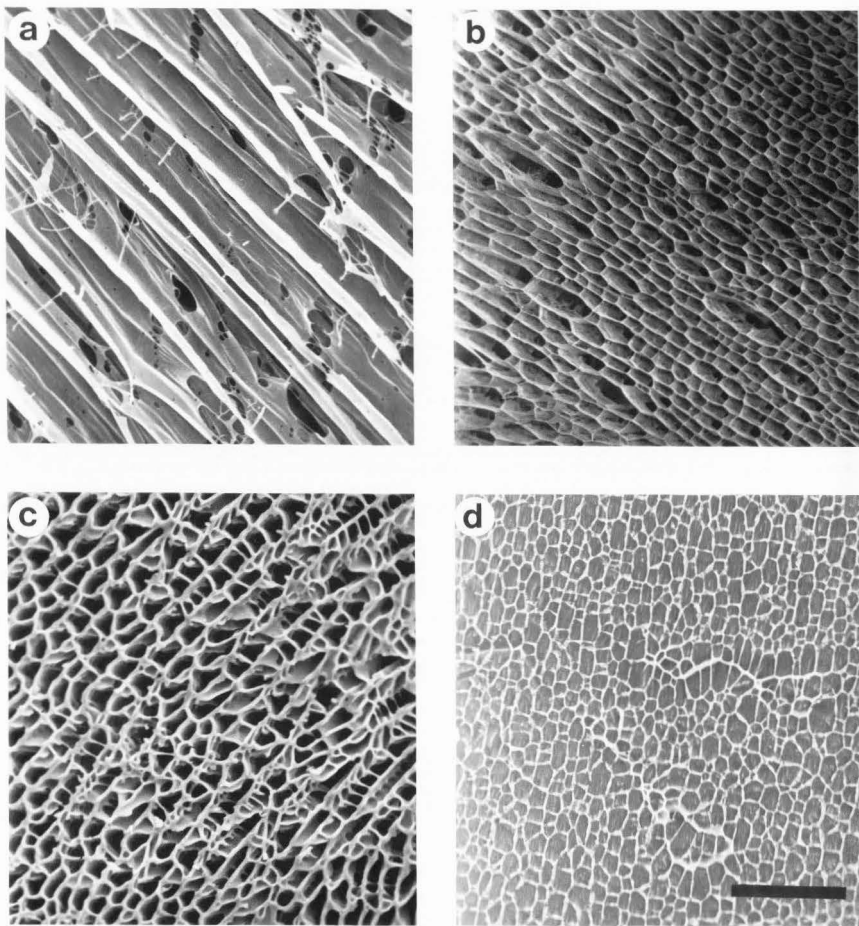


Figure 2. Quench-freezing of 0.2% polysaccharide solutions. a) xanthan; b) locust bean gum; c) carrageenan; and d) guar gum. The ice crystals were parallel to the fracture plane in Fig. 2a, inclined in Fig. 2b, and perpendicular in Figs. 2c and 2d. Bar = 25 μm (all four figures are at same magnification).

two phase structure of ice crystals in a continuous phase that developed as a result of freeze-concentration (Figs. 1b and d). Fig. 1b shows signs of contamination with frost caused by exposure to water vapour during specimen transfer to the microscope (B.E. Brooker, personal

communication). In the 20% solution, the quantity of unfrozen phase resulting from freeze-concentration may not have been sufficient to see the development of the two-phase structure seen in the 40% solution. The water content of a typical ice cream mix more closely

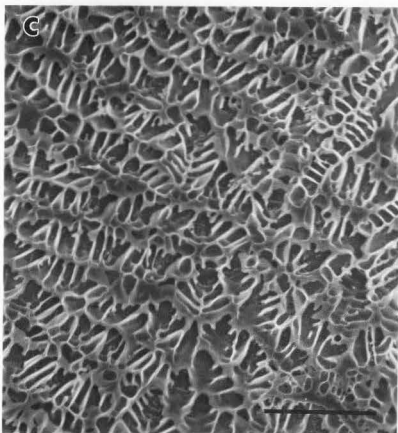
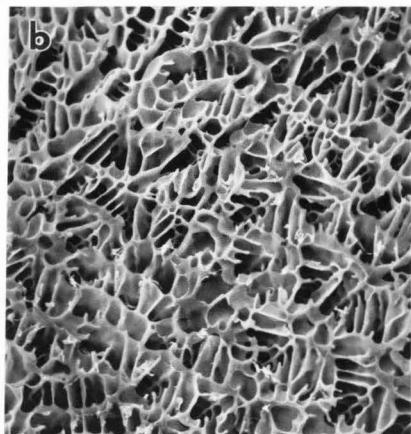


Figure 3. Quench-freezing of 20% sucrose solutions in the presence of 0.2% polysaccharides. a) xanthan gum; b) locust bean gum; c) guar gum. Bar = 25 μm (Figs. 3a, 3b, and 3c are at same magnification).

resembles that of the 40% solution of sucrose, although the freezing point of the solution would be lower than expected in the mix.

Solutions of xanthan gum, locust bean gum, guar

gum, and carrageenan in the presence and absence of 20% sucrose were examined using LT-SEM (Figs. 2 and 3). Polysaccharide stabilizer solutions without sucrose were seen to have a characteristic network structure when quench-frozen during sample preparation (Fig. 2). The network structures that developed were substantial given that the stabilizers were only present at less than 0.5%. It should be noted, however, that these networks may not form in an ice cream mix at the same concentration due to the presence of the many other constituents. The stabilizer solutions that were quenched in liquid nitrogen did not have enough time to freeze concentrate and form large ice crystals; thus the network structures formed were characteristic of the stabilizer present. Xanthan gum microstructure was very fibrous and formed thin honey-comb like strands (Fig. 2a). Locust bean gum appeared to have a more closed honey-comb structure and was very similar to guar gum (Figs. 2b and d). κ -carrageenan (Fig. 2c) tended to form polygonal shapes in the main network with smaller fibrous networks within (Xu *et al.*, 1992).

Quenching of the previous polysaccharide solutions in the presence of sucrose resulted in a similar structure for all solutions and a loss of their characteristic structure (Fig. 3). The presence of sucrose in the stabilizer solutions dominated the network structure that formed. Although the network structure of the polysaccharide was altered by the addition of sucrose, the stabilizers did impart considerable structure to the sucrose solutions which was not seen in their absence (Figs. 1a and 3). The most notable change was the network formed by the

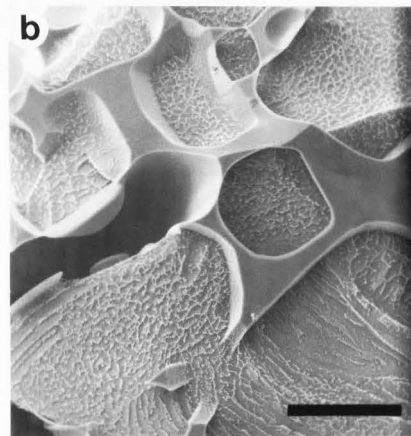
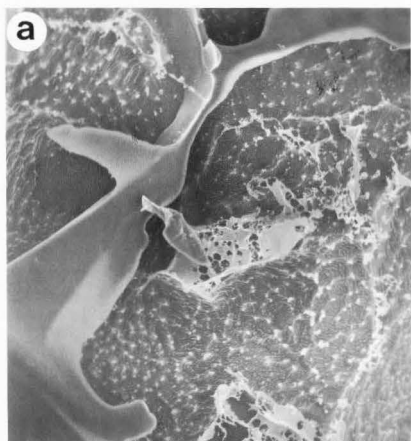


Figure 4. Batch-freezing of guar gum solutions in the presence and absence of sucrose. **a)** 0.6% guar gum; **b)** 20% sucrose plus 0.6% guar gum. Bar = 50 μm (both figures are at same magnification).

xanthan gum in the presence and absence of sucrose (Figs. 2a and 3a).

The polysaccharide/sucrose solutions which were frozen in the batch scraped-surface freezer formed a

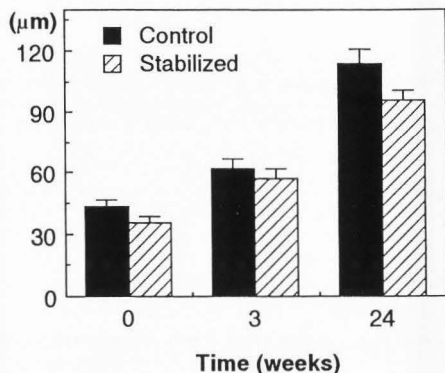


Figure 5. Average maximum diameter (in μm) of ice crystals in stabilized and unstabilized ice creams as a function of storage at heat-shocking temperatures (see text for full description of conditions).

much coarser structure resembling ice cream minus air and fat (Fig. 4). The presence of stabilizer in the sucrose solutions helped the homogeneity of the microstructure due to the enhanced viscosity of the unfrozen phase. The 40% sucrose solution also freeze-concentrated (Fig. 1d), but the microstructure varied from areas of large amounts of ice or solid phases to areas where the ice crystals were obscurely shaped. Strands of material thought to be stabilizer were observed only in the guar gum solution and were found mainly on the ice crystal socket (Fig. 4a).

Ice cream. Microscopic investigation revealed that stabilized ice cream (locust bean gum and carrageenan) had significantly smaller mean ice crystal diameters both initially and as a result of heat shock and storage (24 weeks) compared to those of ice cream without stabilizer (Fig. 5). However, the differences grew larger over time. The influence of the polysaccharide stabilizers on ice cream structure and texture will be discussed further in conjunction with the freezing rate and storage studies.

Influence of Freezing and Hardening Rate

After the initial draw from the continuous freezer, three hardening rates were applied to the product including slow quiescent freezing, plate hardening, and quench freezing. By quenching the ice cream in liquid nitrogen

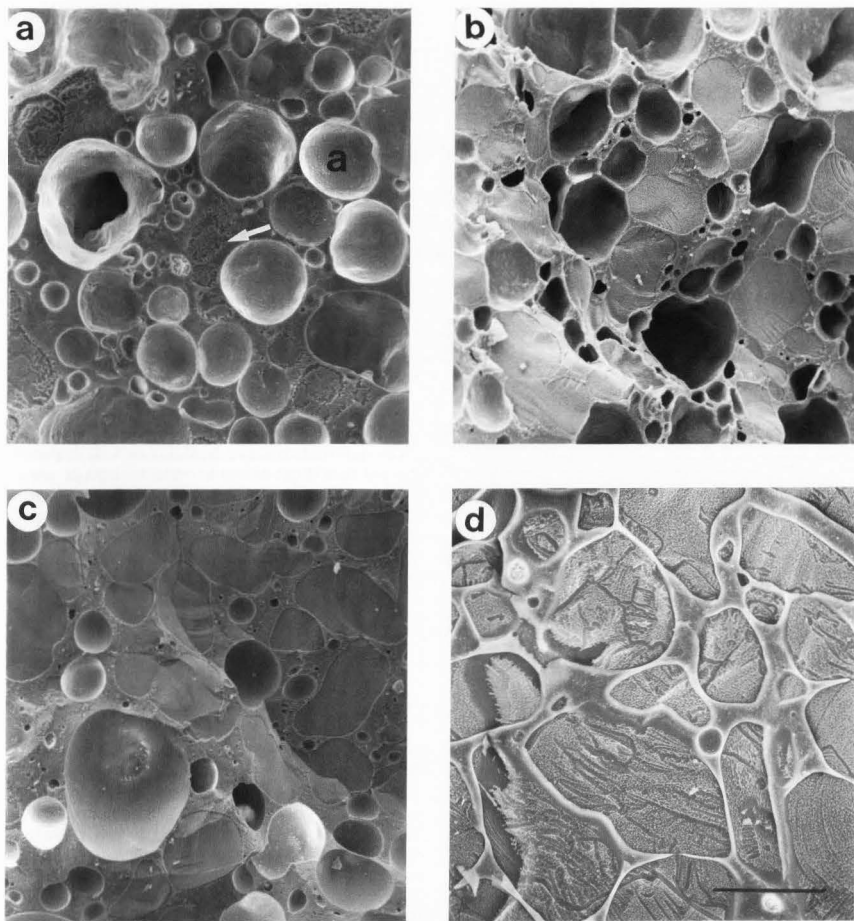


Figure 6. The influence of freezing rate on the microstructure of ice cream. Freezing method: a, b, c) continuous scraped-surface freezing; d) batch scraped-surface freezing. Hardening method: a) liquid nitrogen quench; b) plate freezer, -40°C ; c) and d) hardening room, -25°C . Air bubble (A) and undefined ice crystal borders (arrow) are marked in Fig. 6a. Bar = $50\ \mu\text{m}$ (all four figures are at same magnification).

immediately after draw, the structure formed in the continuous freezer was captured. Ice cream is in a non-equilibrium state as it leaves the continuous freezer. Samples at draw contained a large number of small air

bubbles in the network structure and small ice crystals with poorly defined borders (Fig. 6a). Almost 50% of the fracture surface in the ice cream leaving the freezer was air. The freeze-concentrated serum surrounding the

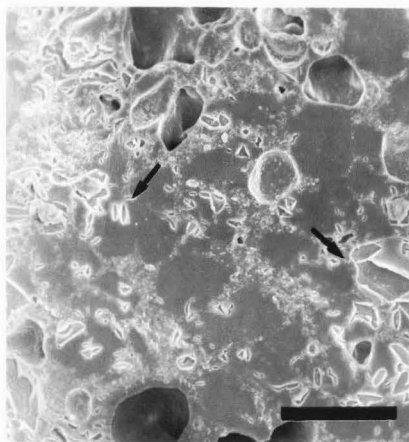


Figure 7. Liquid nitrogen-quenched ice cream after storage at -25°C for one week. Small irregular ice crystals form from recrystallization of solid (vitreous) water (arrows). Bar = $50\ \mu\text{m}$.

Table 1. Means and 95% confidence limits (parentheses) for percentage of total area of micrograph for three ice cream phases as a function of hardening rate and storage time (t). Serum was calculated by difference. The columns for ice cream quench-frozen in liquid nitrogen (LN2) or hardened at -40°C (plate freezer) and -25°C (blast freezer) refer to $t = 0$ weeks. In the last column, ice cream was quench-frozen in liquid nitrogen and then stored at -25°C for two weeks.

Phase	Hardening rate			
	Quenched LN2	Plate -40°C	Freezer -25°C	Quenched 2 Weeks
Ice	17.3 (9.0)	29.1 (5.6)	34.6 (9.7)	33.9 (3.9)
Air	49.4 (9.3)	41.3 (4.9)	25.7 (6.5)	34.0 (5.0)
Serum	33.3 (1.1)	29.6 (2.4)	39.7 (3.2)	32.1 (3.9)

$n = 10$ fields

ice crystals of ice cream had an open structure resulting from the sublimation of solid water present in the serum. In theory, only about 50% of the water present in ice cream mix is in the frozen state at a draw temperature of -5°C (Arbuckle, 1986). The open serum structure and the poorly defined ice crystal borders demonstrated that at draw not all the freezable water was frozen and the freeze-concentration process was incomplete. The water was vitrified or crystallized *in situ* during quenching and did not have time to migrate to the freezing front. When the product was stored for one week or longer at -25°C , small ice crystals were found in the serum phase, indicating a recrystallization of unfrozen water (Fig. 7).

Plate hardening has a slower freezing rate than quenching and more closely represents the freezing rate achieved in industry. The samples quenched in liquid nitrogen were hardened (below -18°C) within a minute, whereas those in the plate freezer required one hour. The microstructure of the ice cream had changed from that of the initial structure indicating that ice cream is not in equilibrium at draw, depending on both temperature and time (Figs. 6a and b). The ice crystals were small, approximately $25\ \mu\text{m}$ in diameter, yet larger than in the quench frozen samples. The ice crystal borders were well defined, suggesting the solutes had more time to migrate away from the freezing front. Micrographs showed a great quantity of air present, but the air bubbles had become larger and many had changed shape to be more like crevices than spherical bubbles (Fig. 6b).

The third and slowest rate of hardening after continuous freezing was accomplished at -25°C . These ice cream samples took at least five hours to become hard (below -18°C). Ice crystal size was larger than the previous hardening rates, having a mean maximum diameter of approximately $35\ \mu\text{m}$ (Fig. 6c). Ice cream hardening rate should be fast enough to allow sufficient ice crystal formation without causing a large degree of foam collapse. The goal of the freezing process should be to create as many small ice crystals as possible. Arbuckle (1986) indicated that ice crystals $> 55\ \mu\text{m}$ were indicative of coarse product. The distribution of ice crystal sizes in the ranges smaller than this benchmark may not be as important for sensory perception. However, if all crystals grow at the same rate, the size distribution would indicate the relative stability of the ice cream to heat shock. Batch freezing followed by quiescent hardening at -25°C produced larger and more irregular ice crystals than continuous freezing (Figs. 6c and d).

The ice, air, and serum phases (by difference) of the ice cream were measured as percentage of total surface area of the micrographs for each of the three hardening rates (Table 1). It takes some time before the ice

phase separates during the freeze concentration process. As the hardening rate became slower, the average area of ice became larger and the average area of air decreased (Table 1). The ice cream hardened in the hardening room had a significantly ($p < 0.05$) larger area of ice than either the plate-frozen or quenched samples (Table 1). Although the total amount of air in the samples was consistent from overrun measurements, it appeared that the smaller air bubbles seen in the freshly extruded product (Fig. 6a) had collapsed and formed larger air bubbles, thus giving the impression of a reduction in surface area of the air bubble cross-section. These observations coincide with the theory that the ice cream is in a non-equilibrium state as it leaves the continuous freezer and more ice is frozen as it is hardened.

Measurements of phase areas were also taken on quenched ice cream after two weeks of storage at -25°C (Table 1). The stored quenched ice cream had significantly ($p < 0.05$) lower air content and significantly ($p < 0.05$) higher ice content compared to freshly quenched ice cream. During quench freezing of ice cream in liquid nitrogen immediately after the draw from the freezer, some water solidified into a glass because there was not sufficient time for crystallization. Thus quenching captures the non-equilibrium state of the product at this stage. However, when the ice cream rewarms to temperatures above its glass transition point, approximately -35°C , the mobility of the constituents increase. The serum changes from a glassy solid to a viscoelastic liquid which is then capable of recrystallizing (Levine and Slade, 1988).

Influence of Storage Times and Temperatures

Both stabilized and unstabilized samples demonstrated ice crystal growth during storage at fluctuating sub-zero temperatures (Fig. 5). Initially, the unstabilized sample had only slightly larger ice crystal sizes and the textures of both samples were acceptable both from sensory perception and based on Arbuckle's (1986) definition of coarse texture (sufficient number of ice crystals greater than $55\ \mu\text{m}$). Figure 8 illustrates the progression of ice crystal growth in the stabilized and unstabilized samples as a function of storage at fluctuating sub-zero temperatures. Control ice creams had more ice crystal fusion profiles than stabilized samples, which also contributed to the increase in ice crystal diameter (Fig. 9). During thermal abuse these merged ice crystals grow as ice melts and water refreezes. The width of the air/serum and ice/serum interfaces in the control ice creams tended to range between 0 (phases touching) and $10\ \mu\text{m}$, whereas stabilized ice creams tended to have larger interfaces ranging from 1 to $25\ \mu\text{m}$. These smaller serum lamellae may be the result of the growth of the ice crystals. The stabilized ice cream had greater

Table 2. Converted mean ranks from the ice cream sensory panel ($n = 46$), analyzed according to the method of Larmond (1977). Heat shocking was performed by moving the samples from -25°C to -10°C for 8 hours daily. A higher mean number corresponds to a coarser sample.

Sample	Converted Rank
Control, stored at -25°C	-0.0243 a
Control, heat shocked	0.7613
Stabilized, stored at -25°C	-0.6270
Stabilized, heat shocked	-0.1100 a

Means followed by the same letter are not significantly different at $p < 0.01$.

amounts of air remaining in the ice cream and more serum interface between the ice crystals. Control product would produce a lower serum viscosity than stabilized product due to the action of the stabilizing gum (Caldwell *et al.*, submitted). Thus, the weaker serum had lower resistance to ice crystal growth and fusion.

Histograms of maximum ice crystal diameter revealed normal distributions in both fresh ice creams (Fig. 10a). Stabilized ice cream had a lower mean and a higher number of smaller ice crystals, whereas unstabilized samples tended to have more ice crystals of large diameter. Minimum ice crystal diameters and ice crystal areas showed similar trends. As both ice creams were aged at fluctuating temperatures, the ice crystals increased in diameter and their distribution became wider. By 24 weeks, the control samples had larger diameter ice crystals than the stabilized samples (Fig. 10c).

Initially, stabilized ice cream showed more air distributed in the fracture surface and the numerous air bubbles had often ruptured at the serum interface (Fig. 11). The control ice creams did not exhibit as many of these ruptures. However, larger air crevices appeared more frequently in control samples and these ice creams were more prone to shrinkage in the package, especially in the heat shocked treatments. Thus stabilized ice cream seemed to hold air that would otherwise be lost in the control ice cream once the air bubble was broken. The greater prevalence of ruptures in stabilized ice cream was attributed to the increased viscosity associated with the serum phases (Caldwell *et al.*, submitted to J. Dairy Sci.) which prevented collapse despite the rupture in the bubble.

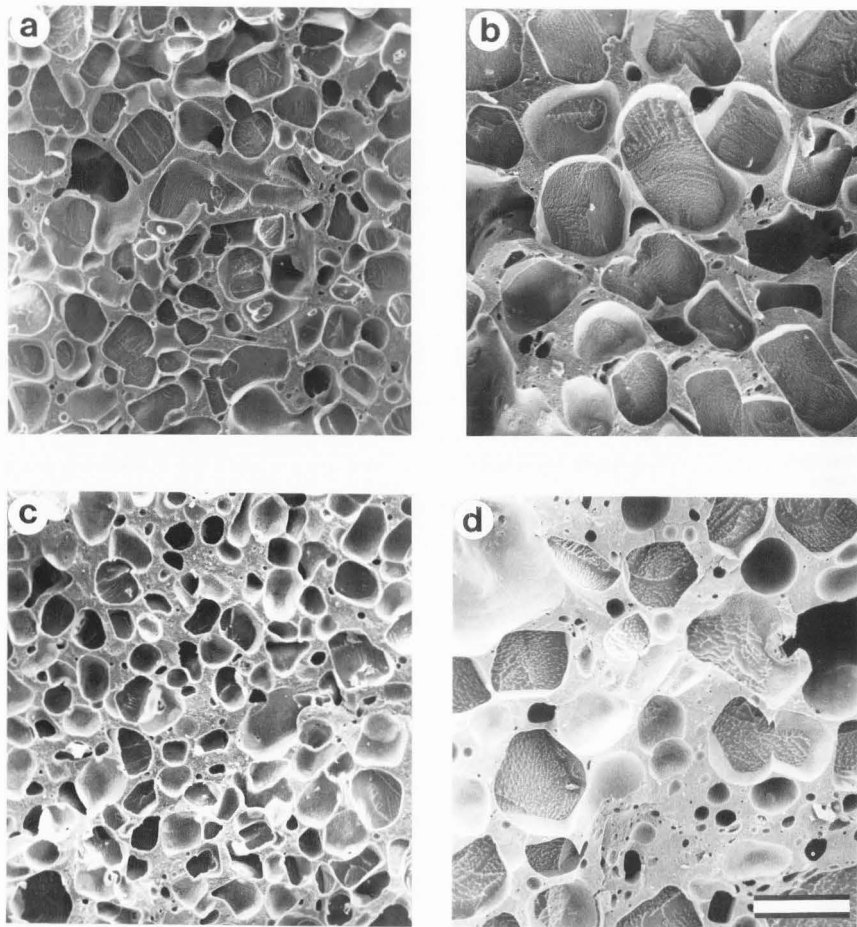


Figure 8. The microstructure of ice cream before and after storage (24 weeks) at temperatures fluctuating between -10°C and -25°C daily. a) and b), control ice cream at 0 and 24 weeks, respectively; c) and d), stabilized ice cream at 0 and 24 weeks, respectively. Bar = $100\ \mu\text{m}$ (all four figures are at same magnification).

Sensory Analyses

The sensory panel on control and stabilized ice cream under unstressed and heat-shock conditions support the data collected on ice crystal size. The sensory

panel results showed a high level ($p < 0.01$) of significant difference between the rankings of the ice creams (Table 2). The heat-shocked unstabilized (control) ice cream was ranked as the iciest sample overall. The

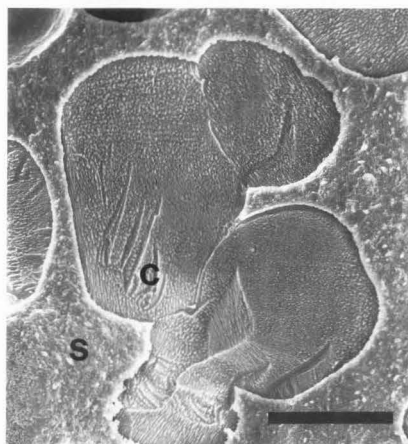
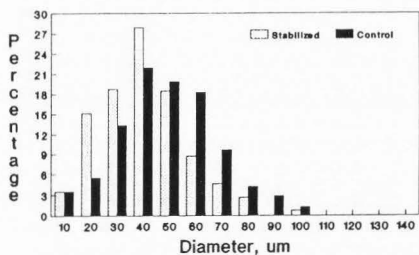


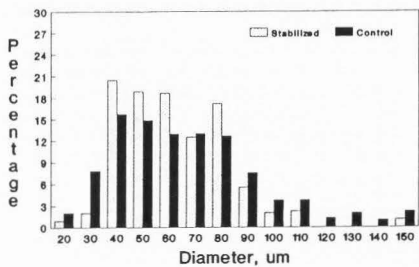
Figure 9 (top). Merged ice crystals (C) surrounded by the serum phase (S) in unstabilized ice cream after 24 weeks. Bar = 25 μ m.

Figure 11 (bottom). Ruptures (arrows) in the air bubble - serum interface in stabilized ice cream. Bar = 10 μ m.

Time = 0 weeks



Time = 3 weeks



Time = 24 weeks

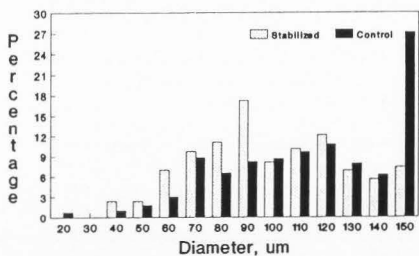


Figure 10. Histogram of maximum ice crystal diameter of control and stabilized ice cream at a) $t = 0$; b) $t = 3$; and c) $t = 24$ weeks at heat-shocking temperatures (see text for full description of conditions).

unstabilized ice cream stored at -25°C and heat-shocked stabilized ice cream did not have significantly different rankings from one another. The least icy sample was the stabilized ice cream stored at -25°C . Even though the ice crystal size data support the findings of the sensory evaluation, Berger *et al.* (1972) feel that subjective assessment is more reliable than objective measurement of ice crystal size because the former takes into consideration other factors influencing the texture such as crystal shape and fat and solids content of the product. The presence of stabilizers can also limit perception of the ice crystals. Many researchers and dairies note that the addition of appropriate stabilizer blends to ice cream produce ice cream with smoother, less icy texture than ice cream of the same formulation without the stabilizer addition. Despite these obvious sensory differences, many researchers have had difficulty in finding explanations for the observed results (Budiaman and Fennema, 1987a, b; Buyong and Fennema, 1988; Muhr *et al.*, 1986).

Conclusions

Polysaccharide stabilizer solutions were seen to have a characteristic network structure when quench-frozen. Although this was altered by the addition of sucrose, the stabilizers did impart structure to sucrose solutions which was not seen in their absence. During batch-freezing of these solutions, the ice crystallization and subsequent freeze concentration process produced ice crystal structures similar to that seen in the microstructure of ice cream (Fig. 4). Examination of different hardening rates of ice cream revealed large influences on microstructure. Quench-hardening did not allow sufficient time for the typical structure of ice cream to develop. The ice crystallization and freeze concentration process had not progressed to the same extent as in products frozen at slower rates. As the hardening rate was reduced, the ice crystals and air bubbles increased in size. It is important to note, however, that the smallest ice crystals and air bubbles were also the least stable over time and thus an optimum freezing rate exists. Pearson (1963) also demonstrated the development of undesirable textures during rapid hardening. However, during batch-freezing or slow hardening, large irregular ice crystals and air crevices were apparent that also led to undesirable texture. Stabilized ice cream initially had smaller mean ice crystal diameters. It also held its structure better during storage at fluctuating temperatures as measured through ice crystal growth and sensory analyses. It appears that the stabilizers increase the resistance/strength of the unfrozen serum phase of frozen ice cream, thus inhibiting mobility and decreasing the degree of recrystallization.

Acknowledgments

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Discussion with Reviewers

R. W. Martin, Jr.: The sizes of the ice crystals in the control ice cream seem exceptionally large and are representative of coarse product. Since the quality of your control is questionable, don't comparisons to experimental variables seem obscure?

Authors: The ice crystal sizes of the control ice cream are larger than preferred industrial production. The ice cream was produced on a pilot plant scale and hardened in a quiescent freezer at -25 °C. Hardening took several hours. However, it was not considered coarse, either by sensory grading or by definition of Arbuckle (1988), i.e., below the limit of 55 μm . Initially, both the control and stabilized ice creams were of acceptable quality. Although the data suggested that the stabilized ice cream started off with a smaller mean ice crystal size distribution, the differences were small. However, during the heat-shock treatment, the differences became larger and thus we are confident of the results despite the size of the initial ice crystals.

D. F. Lewis: How do you distinguish between touching ice crystals? For example, on Fig. 6c, would you count the feature below the label as one or two crystals?

Authors: A general rule used in distinguishing between touching and merged crystals was that if at least 2/3 of the side of the crystals were adjoining, then it was considered as one crystal, and approximate measurements of the longest and shortest axes (maximum and minimum diameters) and the perimeter were taken. This was subjective but care was taken to be as consistent as possible throughout. Thus the structure in Fig. 6c would be measured as one crystal.

D. F. Lewis: You will be measuring ice crystal size more or less at a single plane through each crystal. Consequently you will have two size distributions superimposed on each other - one due to "real" variations in crystal size and the other a natural effect of seeing different crystal planes in similar sized crystals. Do you have a philosophy to allow for these two effects?

Authors: Although the authors recognize the difficulty which is expressed in this question, it was felt that by treating each measurement as consistently as possible, by treating all fields randomly (i.e., not trying to force an orientation to a crystal), and by having a large sample size, any bias from orientation effects would be minimized. During sublimation of the crystals, it is noted that the crystal size did not appear to change in dimension due to increased etching (Caldwell *et al.*, 1992). Thus small changes in the measured maximum diameter of the ice crystal did not greatly influence the overall sample mean. Ice cream samples have been measured by us using both the SEM and light microscopy (Unpublished data). The sample mean maximum diameters were within 2 μm of each other. Light microscopy measures the largest diameter regardless of the orientation of the crystal. The SEM measures the maximum diameter at the surface plane. The differences were insignificant.

O. Johari: Please provide additional information about the Caldwell *et al.* submitted reference.

Authors: The paper "Caldwell KB, Goff HD, Maurice TJ. The influence of polysaccharides on glass transitions and low-temperature stability of ice cream" has been submitted to *J. Dairy Sci.* A copy is available from H.D. Goff (address on page 11).