

Influence of emulsifiers on ice cream produced by conventional freezing and low-temperature extrusion processing

S. Bolliger^{a,b,1}, B. Kornbrust^{a,c}, H.D. Goff^{a,*}, B.W. Tharp^b, E.J. Windhab^c

^aDepartment of Food Science, University of Guelph, Guelph, Ont., Canada N1G 2W1

^bGermantown International Ltd., 200 Lawrence Drive, West Chester, PA 19380, USA

^cFederal Institute of Technology (ETH), Laboratory of Food Engineering, Schmelzbergstrasse 9, CH-8092 Zurich, Switzerland

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Abstract

Ice cream at six different levels of emulsification was produced by freezing in a conventional scraped surface freezer and with a serial configuration of a conventional freezer followed by a low-temperature extruder. The aim was to examine the influence of emulsifiers on the process, since both emulsifier addition and low-temperature extrusion may have similar effects on promotion of colloidal structure in ice cream. Ice cream samples from both processes were analyzed for stiffness at draw, fat destabilization indices, melting performance, and microstructure. Low-temperature extrusion generally promoted enhanced fat destabilization, however, fat particle size and solvent extractable fat showed different dependencies on emulsification level from the two processing systems. Although solvent extractable fat reached high levels with increasing emulsification, fat particle size data suggested that fat agglomerate size was controlled by mechanical shearing. Significant difference between the two systems was seen also in the meltdown test, where melting rates for unemulsified and slightly emulsified mixes led to a very low melting rate and high shape retention in extruded ice cream. Scanning electron microscopy detected generally smaller air bubbles for extruded ice creams. Enhanced fat structuring around the air bubble and into the serum phase was also shown for unemulsified extruded samples. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Ice cream processing; Low-temperature extrusion; Fat destabilization; Emulsifier functionality; Ice cream structure

1. Introduction

Conventional ice cream freezers are scraped surface heat exchangers with high dasher rotational speed and attached surface-scraping knives. The conditions in this heat exchanger favor good heat transfer as well as a relatively efficient way of aerating the product, but are limited in their attainable draw temperature. The most common draw temperatures for conventional freezers are -5 to -6°C , with some systems achieving temperatures down to -9°C . However, a low-temperature freezing extruder now available takes ice cream from conventional continuous freezers and further freezes it to

draw temperatures below -15°C , under low shear conditions, at $200\text{--}2000\text{ L h}^{-1}$ ice cream. This mechanical treatment at high ice crystal volume fraction and high viscosity reduces the size of dispersed phase ice cream components, i.e. ice crystals, gas bubbles and agglomerated fat globules (Windhab & Bolliger, 1998a, b). Systematic investigations of ice cream in rheometric shear gaps at low-temperatures provided quantitative relationships between structural changes of the dispersed components, energy dissipation and related temperature increase and remelting. The main optimization characteristics of the resulting low-temperature ice cream extrusion system (Windhab & Bolliger, 1998a) were: (a) uniformity of local shear stresses acting in the extrusion channel flow; (b) maximum structure development of the dispersed phases and good mixing without local remelting; (c) maximum heat transfer coefficient. This work addresses the second of these important optimization requirements.

A study of the influence of extruder parameter settings on ice crystal size in stabilized sucrose solutions (Bolliger,

* Corresponding author. Tel.: + 1-519-824-4120, ext. 3878; fax: + 1-519-824-6631.

E-mail address: dgoff@uoguelph.ca (H.D. Goff).

¹ Present address: Centre R & D Nestlé, Rue Charles Tellier, F-60000 Beauvais, France

1996) showed that rpm was inversely correlated with median ice crystal size $d_{50,0}$ when overrun was set to 60%. Furthermore, an increase in overrun had a retarding effect on ice crystal growth during storage. Windhab and Bolliger (1998a, b) produced data regarding the influence of the extrusion system on fat structure parameters such as fat agglomerate size, solvent extractable fat, fat agglomeration index and melting rate. For ice cream products (10% fat content) obtained at two levels of overrun, solvent extractable fat percentages were 14.8% (freezer) and 18.1% (extruder) for 120% overrun and 8.6% (freezer) and 23.2% (extruder) for an overrun of 55%. Hence, the extruded ice creams showed higher levels of solvent extractable fat when compared to conventional frozen ice creams. A sensory test showed that extruded ice cream was felt less hard and less cold. No data regarding fat agglomerate size were given. It was postulated that during cold extrusion, fat globules are sheared to platelet-type structures. It was also noted that for highly viscous systems at temperatures below -13°C , large fat agglomerates were not formed. Hence, a buttery mouthfeel was not created. Air bubble distributions for freezer and extruder ice creams (10% fat content) showed that in extruded ice creams the size distribution shifted to smaller air bubbles, compared to conventionally frozen ice cream. It is also known that processing parameters increasing the shear that mix is subjected to in the freezer (i.e., draw temperature, pressure) will increase fat agglomeration (Kokubo, Sakurai, Hakamata, Tomita, & Yoshida, 1996; Kokubo, Sakurai, Iwaki, Tomita, & Yoshida, 1998). In a paper discussing pre-aeration for conventional freezing, Windhab and Bolliger (1995) concluded that a finer air bubble distribution can be achieved with pre-aeration. However, mechanical over-stressing during the freezer stage could cancel out this advantage. According to a patent for low-temperature extrusion processing of ice cream (Windhab, Fels, Hoffmann, & von Holdt, 1993), the extruder is claimed to provide a good uniformity in shear stresses without exceeding critical levels causing remelting. Thus, pre-aeration could have potential advantages for extrusion technology.

The colloidal structure and action of emulsifiers in ice cream have been reviewed (Goff, 1997). Briefly, emulsifiers improve shape retention during extrusion and during meltdown by inducing partial coalescence of the fat during freezing. The fat aggregates thus formed are capable of building structure into the unfrozen phase and stabilizing the air bubbles (Goff, Verespej, & Smith, 1999). In this work, the difference between conventional freezing and low-temperature extrusion technology was further investigated, with the aim of examining in particular the influence of emulsifiers on the process, since both emulsifier addition and low-temperature extrusion may have similar effects on promotion of colloidal structure in ice cream.

2. Materials and methods

2.1. Mix and ice cream preparation

Six different ice cream recipes were produced, following a similar production protocol to that used by Bolliger, Tharp, and Goff (2000). All mixes contained 10% milk fat (anhydrous milk fat, Gay Lea, Guelph, Canada), 10% milk solids-not-fat (skim milk powder, Ault Foods, Mitchell, Canada), 12% sucrose (Lantic Sugar Limited, Toronto, Canada), 6% corn syrup solids (Dry Sweet 42, Roquette, Keokuk, USA), and 0.15% stabilizer (guar gum, Germantown, Toronto, Canada). The emulsifier content of the mixes was varied as follows: no emulsifier; 0.075% mono- and di-glycerides (mdg), (40–42% α mono-glyceride, iodine value <3 , Germantown Toronto, Canada); 0.15% mdg; 0.15% mdg + 0.02% polysorbate 80 (ps80), (Germantown, Toronto, Canada); 0.15% mdg + 0.04% ps80; 0.15% mdg + 0.06% ps80. All experiments (production and analyses) were carried out in triplicate.

Raw ingredients were shipped dry or frozen from Canada to ETH Laboratory of Food Engineering, Zurich, Switzerland, for ice cream manufacture. All ingredients except anhydrous milk fat were dry blended, mixed with water, and immediately blended with the milk fat portion. Mixes were pasteurized at 70°C for 20 min and homogenized with a single-stage homogenizer (Rannie) in two passes, first in a high-pressure step at 17.5 MPa and then in a low-pressure step at 3.5 MPa. The mix was then aged at 4°C for 24 h.

A combination of a traditional ice cream freezer and a low-temperature extruder was used for the ice-cream production. The conventional freezer (Model C, APV Crepaco) was used as the first cooling step and operated in a serial configuration with the low-temperature extruder (Model 65-100, Schroder GmbH and Co., Lubeck, Germany). The freezer exit was connected with the extruder entrance using a double jacketed (-5°C) pipe. Partly frozen and aerated ice cream was released from the freezer and continuously transferred into the extruder without a temperature increase. In the extruder further freezing and structuring took place. All necessary process control data (volume flow, pressure and temperature at entrance and exit of freezer and extruder) were registered on-line. The production equipment (freezer/extruder) was always precooled by freezing a sucrose solution (25% sucrose, 0.5% stabilizer; held at 4°C). Therefore, the target temperature of -4.5°C freezer exit and -13.5°C extruder exit was established faster. The extruder speed was set to 20 rpm and overrun to 100% at a mix flow rate of 45 L h^{-1} . The process was defined as stable when the target parameters were obtained as closely as possible and the measured values showed only a small fluctuation over a period of 10 min. The average exit temperature from the freezer was $-4.4 \pm 0.4^{\circ}\text{C}$, and the average exit

temperature from the extruder was $-13.3 \pm 0.3^\circ\text{C}$. Eight ice cream samples (375 g) were then taken, starting with the extruder and continuing with the freezer ice cream, and all samples were stored at -28°C for hardening.

2.2. Analyses

Stiffness at draw: Firmness of the ice cream samples at the time of draw from the freezer and the extruder was measured by penetration of a 2 cm diameter cylinder into the ice cream to a depth of 4 mm at a constant speed of 2 mm s^{-1} with a Texture Analyzer (TAXT2, Stable Microsystems, Kent, England).

Fat particle size analysis: Particle size distribution of the melted ice creams (4°C for 3–4 h) after hardening was measured by integrated laser light scattering, using a Mastersizer X (45 mm lens, manufacturer's presentation code 0303, Malvern Instruments Ltd., Malvern, Worcs., UK). The measurements were carried out at room temperature. The dilution of the emulsion in the sample chamber was approximately 1:1000. Mean particle size diameter $d_{4,3}$ (the volume-weighted mean diameter) was recorded.

Fat agglomeration index: To evaluate the degree of fat destabilization by spectroturbidity (Goff & Jordan, 1989), hardened ice cream samples were thawed and an aliquot of 40 mL was taken for analysis. Mix and ice cream samples were diluted 1:500 with deionized water and absorbance was measured by a spectrophotometer (Lambda 2 UV/VIS-Spectrometer, Perkin-Elmer, Switzerland) at 540 nm against deionized water as a blank. Three measurements per dilution were carried out. Fat agglomeration index was calculated as (absorbance in mix – absorbance in ice cream)/absorbance in mix $\times 100\%$.

Melting test: Samples of ice cream after hardening from both processes were subjected to a heat shock of -15°C for 6 h, -5°C for 6 h, for 12 days in a temperature programmable cabinet (Scientemp Model 34-23, Chicago, IL). Ice cream samples before and after heat shocking were subjected to a quantitative melt down test. All the samples were tempered at -22°C . Samples for the meltdown test (260 g) were liberated from the containers and weighed on a scale. The samples were placed on a 10 mesh grid ($= 10\text{ holes } 2.54\text{ cm}^{-1}$) and allowed to stand at ambient temperature (20°C). The weight of the material passing through the screen (referred to throughout as dripped portion) was recorded after 20, 30, 50, 60, 70, 100, 110, 120 min. The remaining portion after 120 min was quantitatively removed, weighed and both fractions were analyzed for total fat and protein. The melting rate (slope) was determined based on graphs of melted portion as a function of time (values before 40 min were omitted). The original mix and fractions (dripped and remaining portion) from the melting test were analyzed

for fat by Roese-Gottlieb analysis (Eidgenössische Forschungsanstalt für Milchwirtschaft, Liebefeld, Switzerland).

Solvent extractable fat (SEF): The analysis of SEF was carried out at Laboratory Services, University of Guelph, Canada. Packaged, hardened ice cream samples were shipped by air freight, overnight on dry ice, from Switzerland to Canada. The ice cream temperature was less than -30°C upon receipt, and was stored at -35°C until analyses. Heptane (Fisher Chemicals, H350, HPLC grade) was used as a solvent in order to partially extract fat from melted ice cream samples. Prior to the test, the samples were stored at -22°C . About 25 g of the frozen sample was weighed into a 125 mL separation flask. Samples were thawed for 2 h at 3°C . After thawing, 30 mL of heptane was added to each sample. The separation flasks were rotated at 180 rpm for 30 s. After a 3 min pause this treatment was repeated. Subsequently, the separation flasks sat vertically for 60 min in order to attain adequate separation. The heptane phase was then transferred into a conical flask by means of a 10 mL pipette. The extraction, starting with the addition of 30 mL of heptane, was repeated two more times (always with 60 min standing time). For the second and third extraction steps, sample rotation ($180\text{ rpm } 30\text{ s}^{-1}$) was only performed once. Samples were kept in a fume hood until visually determined as dry, and the conical flasks were then dried at 105°C for 4 h. They were transferred to the sample dry keeper for 30 min and weighed. The percentage of solvent extractable fat was calculated.

Scanning electron microscopy (SEM): SEM analysis was conducted on hardened ice cream samples after heat shock, as above, at the University of Guelph. After temperature cycling was completed, small samples (approx. $5\text{--}6\text{ mm}^3$) were immersed in liquid nitrogen (-196°C) and were carefully fractured with a slight tap and reduced to smaller pieces while still immersed in liquid nitrogen. Small chips were collected and stored in cryo-vials in liquid nitrogen until preparation for the cold stage in the Hitachi S-570 SEM (Hitachi Ltd., Tokyo, Japan). The sample stub for cryo-SEM consisted of a copper base with a gorged area along the stub and two spring-loaded supports. Two specimens ($2\text{--}3\text{ mm}^3$) were placed in the stub while immersed in liquid nitrogen slush. The stub was then transferred at a reduced pressure of 1×10^{-2} Torr to the preparation chamber (EMscope SP2000A Sputter-Cryo Cryogenic preparation System, EMscope Ltd., England) using the transfer device. The specimens were fractured using the blade in the preparation chamber and then transferred to the cold stage of the microscope for sublimation, to eliminate surface frost and clearly outline the perimeter of ice crystals within the unfrozen phase (15 min). After etching, the stubs were transferred back to the preparation chamber for gold sputter coating (20–30 nm). Specimens were viewed at 10 kV accelerating voltage with an objective lens aperture

of 50 μm . Photographs were collected using the Mamiya ProSD-120 (Camera Co. Ltd., Japan) and Illford FP4-125 film.

3. Results and discussion

The low-temperature extruder was freezing ice cream from -4.5°C , after draw from a conventional freezing process, to -13.5°C under low-shear conditions within the twin-screw configuration. Ice creams produced both from low-temperature extrusion and from conventional hardening had been continuously frozen through the same scraped-surface equipment to a draw temperature of -4.5°C . Thus, the effects of low-temperature extrusion should be considered as additive to or modifying the structure initially created in the conventional scraped surface freezer. The stiffness at draw is shown in Fig. 1. The overwhelming influence on the stiffness is the ice content, as the comparison here is between ice creams at -4.5 and -13.5°C . Two important observations emerge. One is that the low-temperature extruded ice cream is considerably firmer, as expected, and this will have a great impact on subsequent unit operations such as ingredient addition, shape-forming, packaging, etc. The second observation concerns the emulsifier addition. After conventional freezing, the emulsifier increases the stiffness at draw, due to its effect on fat structuring and also possibly on air bubble size distribution. However, after low-temperature extrusion, there is less influence of

emulsifier addition on stiffness, with the high ice content overwhelming and masking the effect of fat and air structures. The non-emulsified mix remains the lowest but not significantly different in stiffness, and there is no significant difference with either emulsifier type or concentration. Constant rheological characteristics at draw are a prerequisite for good production control, where often many after-forming and packaging operations are dependent on the stiffness at draw.

An effect of the further application of shear during low-temperature extrusion might be expected on the extent of fat structuring, as shear is known to influence fat destabilization (Kokubo et al., 1996, 1998). Emulsifiers are known to promote enhanced fat structuring, and thus an effect of emulsifier addition after low-temperature extrusion might also be expected. The results of fat agglomeration index, solvent extractable fat, and fat aggregate size measurements are shown in Fig. 1. It should be noted that actual size distributions were bimodal, with a second peak of aggregates of 30–40- μm size increasing in frequency as the extent of emulsification or application of shear increased (Goff, 1997). Thus, a mean particle diameter only provides an indication of the shifting size distribution. Several observations can be made. The results from conventional freezing are as expected and mirror those obtained previously (Bolliger et al., 2000): an increase in fat destabilization measurements with increasing emulsifier concentration, and a noticeable jump with the addition of polysorbate 80. All measurements of fat destabilization indicated enhanced levels after low-temperature extrusion. It was also seen that solvent extractable fat increased continuously as a function of the emulsification level. The linear correlation coefficient (r^2) between solvent extractable fat values from conventional freezing and low-temperature extrusion processing was 0.97. However, with fat agglomeration index and fat agglomerate size, a slightly different functional dependency could be seen. At higher levels of emulsification, fat agglomeration index and fat agglomerate size were less affected than solvent extractable fat by the low-temperature extrusion. The linear correlation coefficient (r^2) between fat agglomeration index values from conventional freezing and low-temperature extrusion processing was only 0.51.

It appears that the extruder was increasing the extent of fat agglomerates formed, but that the size of the agglomerates was being controlled to a maximum level by shearing action in the extruder. Based on the physical phenomena being measured by these indices of fat destabilization (Goff, 1997; Goff et al., 1999), an increased proportion of aggregates of smaller size would have more influence on solvent extractable fat measurements, while an increasing agglomerate size with smaller numbers would have more influence on turbidity and volume-weighted diameter. The actual size distributions were bimodal, with the second peak of aggregates forming at

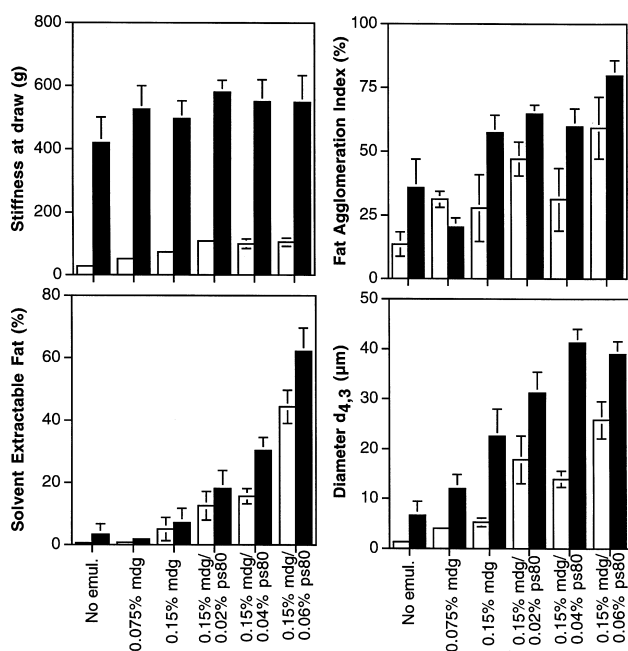


Fig. 1. Physical parameters of ice cream at draw from freezer (white bars) and extruder (black bars) as a function of the emulsification level. Error bars are standard deviations; $n = 3$.

30–40 μm and remaining constant in size, but showing increased proportions (height) with increasing emulsifier concentration. Hence, the increased solvent extractable fat measurements with low-temperature extrusion may not accurately reflect the effect of this process on the structure of the ice cream. From a processing point of view, this means that the extruder will lead to further agglomeration for poorly emulsified systems; however, it will not lead to excessive agglomeration of strongly emulsified mixes. A limitation on maximum agglomerate size in the extruder process was also proposed by Windhab and Bolliger (1998a, b); butteriness in extruder products did not appear because agglomerates were not allowed to grow large enough, due to the shearing action of fat globule agglomerates in the highly viscous system, at temperatures below -13°C .

Based on the results of Bolliger et al. (2000), it seemed also informative to examine quantitatively and qualitatively the meltdown of the ice cream produced. An assumption is made that the rate of melting of ice was similar in all ice creams, both conventionally frozen and low-temperature extruded, as dictated by heat transfer between the environment and the sample, which was similar in all cases. It may be possible that differing air bubble distributions had an impact on the melting of ice, but it seems more probable that melting rates at the end of 120 min were influenced mostly by fat structuring. A remarkable difference in melting rates between freezer and extruder ice cream was observed (Fig. 2). While a change in emulsification levels for conventionally frozen ice cream led to a rather continuous and steady reduction in melting rates, only a very small change between non-emulsified and fully-emulsified ice creams was noticed for the low-temperature extruded samples. This relationship was confirmed when ice cream submitted to heat shock was measured. This heat shock treatment was performed to simulate temperature-fluctuating

storage/distribution conditions, and thus would be an indication of structural stability. Non-emulsified ice creams and those with mdg only showed the largest increases in melting rate after heat shock for conventionally frozen and hardened ice cream, whereas for the extruder only the unemulsified ice cream showed a significant increase in melting rate due to the heat shock (data not shown). This would be indication of improved stability due to structure formation.

As seen in Fig. 2, total fat analyzed in the melted portion from the melting test was higher when produced in the freezer, especially for the mixes containing no polysorbate 80. This is an indirect indication of the existence of a richer fat phase in the remaining portion, and in a previous publication (Bolliger et al., 2000), fat in the dripped portion of a meltdown test from conventionally frozen ice cream was found to correlate well with fat destabilization measurements. The slower melt down of extruder ice cream was parallel with the observed increase of fat in the remaining portion from the melting test. This relates well to the fat destabilization measurements found here and provides further evidence of an enhanced fat network after low-temperature extrusion that stabilizes the ice cream structure during melt down.

Since melting rates are less a measure of actual physical melting of ice than an indication of structure stability (collapse and flow of the fat structure after the melting of ice), it is relevant to complete the information with a documentation of shapes (Fig. 3). Ice creams after heat shock were melted and images were taken in regular intervals. All low-temperature extrusion samples showed significantly greater shape retention than conventionally frozen ice creams, regardless of emulsifier level. The shape retention evaluations allow for enhanced interpretation of the fluid loss (melting rate) data. Although low-temperature extrusion samples for the non-emulsified mix shows a low degree of fluid loss (Fig. 2), the structure and shape of the ice cream sample changed significantly, indicating less fat structure retention than in the emulsified samples. However, for the lowest level of emulsifier (0.075% mdg) more structural stability from low-temperature extrusion was shown compared to the conventional freezer. For 0.15% mdg only a slight increase in the shape retention of the freezer sample was noted. The extruded sample showed virtually complete shape retention, without any influence of polysorbate 80. Beginning with the addition of polysorbate 80, the freezer sample showed signs of an agglomerated fat structure influence, since the shape showed good retention. The extruded sample showed complete shape retention. Shape retention images from the conventional freezer samples with 0.15% mdg and 0.06% polysorbate 80 were comparable to those with no polysorbate 80 from the low-temperature extruder, suggesting that the low-temperature extrusion process provided properties at low

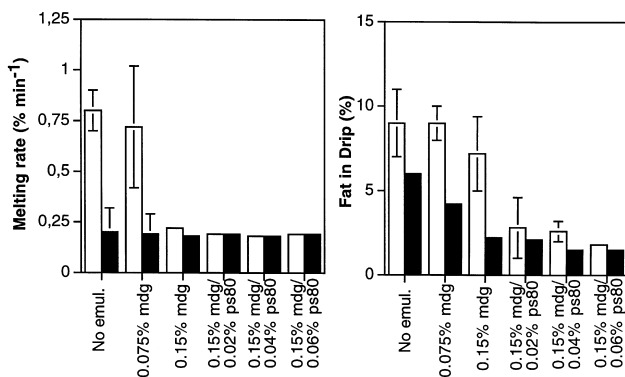


Fig. 2. Performance during the melting test of hardened ice cream from freezer and conventional hardening (white bars) or low-temperature extruder (black bars) as a function of the emulsification level. Error bars are standard deviations; $n = 3$.

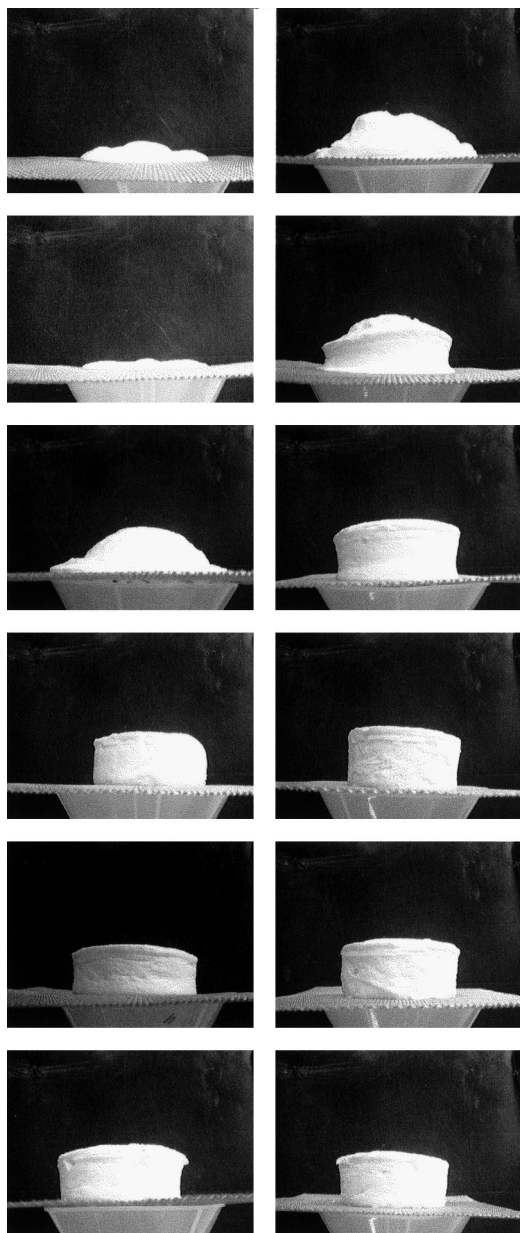


Fig. 3. Shape retention after 120 min during the meltdown test of ice cream frozen in the continuous freezer followed by conventional hardening (left side) or with the low-temperature extruder (right side), both after heat shock (– 5 to – 15°C temperature cycles, twice daily for 12 days), as a function of emulsification level. From top to bottom, the ice cream emulsifier composition was: no emulsifier; 0.075% mono- and di-glyceride; 0.15% mono- and di-glyceride; 0.15% mono- and di-glyceride plus 0.02% polysorbate 80; 0.15% mono- and di-glyceride plus 0.04% polysorbate 80; 0.15% mono- and di-glyceride plus 0.06% polysorbate 80.

levels of emulsification matching those of highly emulsified conventionally frozen ice creams.

Some explanation for the good shape retention and melting rate properties in the extruded samples was found by a comparison of cryo-SEM images (Fig. 4).

Extruded samples show a relatively fine air bubble distribution (Fig. 4(B1–B3)) with many bubbles in the 10–15 µm range, compared to conventionally frozen ice cream samples (Fig. 4(A1–A3)) with bubbles in the 40–70 µm range. However, only the conventional freezer samples showed a qualitative reduction of air bubble diameters (Fig. 4(A1–A3)) when the level of emulsification increased. Extruded ice creams, at least on a qualitative basis, did not change in air bubble diameter (Fig. 4(B1–B3)). The fat and air structures observed in the non-emulsified low-temperature extruded ice cream were more similar to slightly emulsified samples from conventional processing reported earlier (Goff et al., 1999; Bolliger et al., 2000).

Hence, it is postulated that in non-emulsified ice creams or ice creams formulated with low concentration of emulsifiers, the smaller air bubble structure and influence of the additional mechanical action on fat destabilization helps to stabilize ice cream structure during meltdown and leads to the shape retention characteristics observed. Mechanical-induced separation of air bubbles is expected to occur to a greater extent in the extruder, and due to the low-temperatures in the extruder, the smaller air bubble distribution is stabilized simply with the aid of viscous stabilization by a continuous ice cream matrix of high viscosity. Hence, the yield stress necessary to move the continuous ice cream system that is around the air bubbles and ice crystals cannot be exceeded by the Laplace pressure of the single air bubbles, leading to high stability of the air bubbles.

4. Conclusions

Ice cream produced in low-temperature extrusion equipment behaved differently with respect to emulsification properties compared to conventional processing. Indices of fat destabilization suggested that the extruder promoted fat structuring with no or low levels of emulsifier, but did not lead to excessive formation of agglomeration for highly emulsified mixes, typical of those with added polysorbate 80. Ice creams with no or low concentration of emulsifiers are pushed to a higher level of agglomeration, which in return is partially responsible for low melting rates and good shape retention. In the case of no or low concentration of emulsifiers, extruded ice creams behaved significantly better in the melting test and showed significantly finer air bubble distributions and more fat structuring around the air phase. This work thus provides further information about the complex relationship between processing and ingredient performance when assessing process modifications such as low-temperature extrusion processing. It is apparent that formulation modification and optimization must also occur.

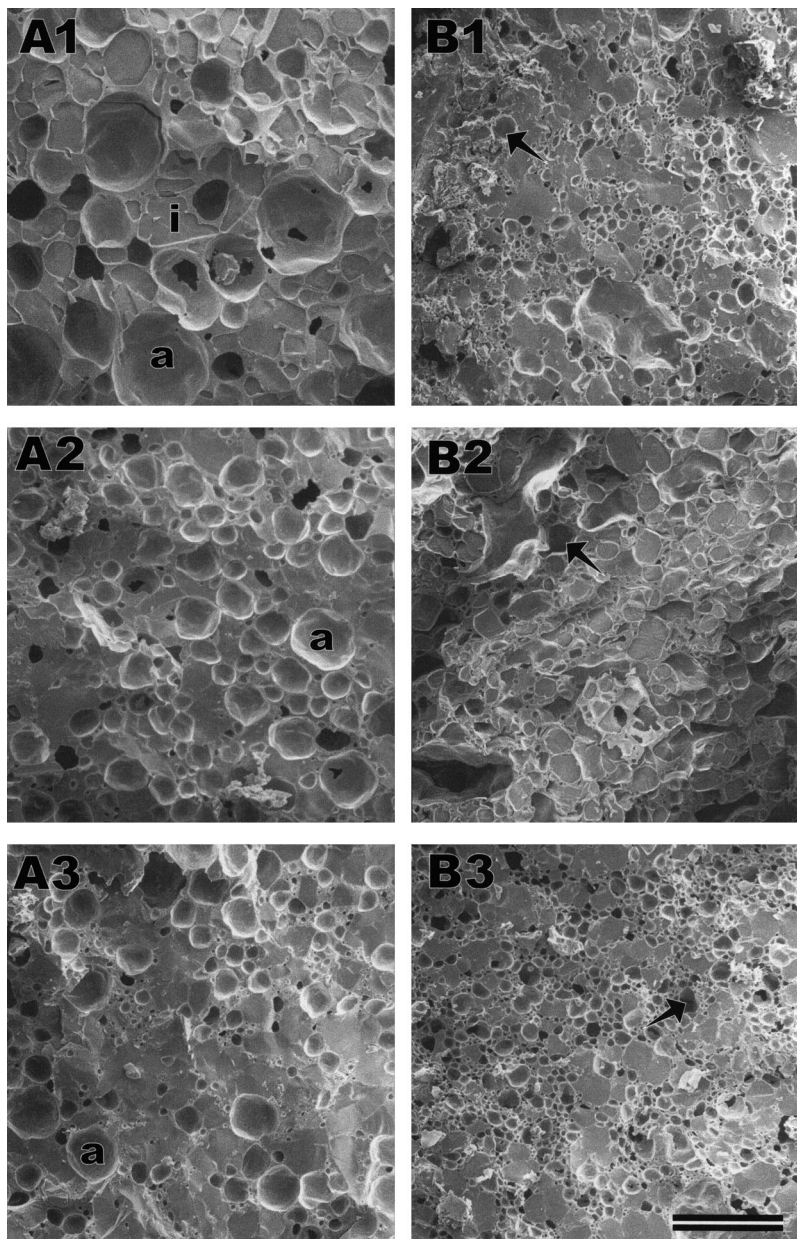


Fig. 4. Cryo-SEM images of hardened ice cream produced with no emulsifier (1), 0.15% mono- and di-glycerides (2), and 0.15% mono- and di-glyceride plus 0.06% polysorbate 80 (3): by means of a freezer (A) and an extruder (B); a or arrowhead = air bubble, i = ice crystal. All images are at the same magnification; bar = 200 μm .

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