

Correlation between colloidal properties of ice cream mix and ice cream

S. Bolliger^{a,b,1}, H.D. Goff^{a,*}, B.W. Tharp^b

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

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

Received 5 November 1999; accepted 14 April 2000

Abstract

Ice cream mix was produced with a range of emulsifiers and concentrations. Ice cream mix properties were measured and correlated to ice cream properties. Protein load (mg m^{-2}) 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). Thus, the measurement of protein load in the mix can be used to predict ice cream fat stability and related structure with constant processing conditions. As emulsification increased, more fat could be seen at the air interface by scanning electron microscopy. High correlation coefficients were also obtained with fat structure analyses and the quantitative determination of fat in the dripped portion taken from a melting test of ice cream. Hence, fat analysis from the dripped melt fraction is suggested as a method to characterize the fat-related structure in ice cream. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Ice cream; Fat destabilization; Partial coalescence; Solvent extractable fat; Electron microscopy; Emulsifier; Meltdown

1. Introduction

Fat contributes greatly to the structure of ice cream during freezing and whipping by forming a partially coalesced three-dimensional network of homogenized globules that, along with air bubbles and ice crystals, is responsible for stiffness and dryness in the extruded product, as well as melt resistance and smoother texture in the frozen product (Berger, 1997; Goff, 1997; Campbell & Pelan, 1998; Tharp, Forrest, Swan, Dunning & Hil-moe, 1998). Fat globules, either discrete or partially coalesced, are found both at the air interface and in the serum phase of the ice cream (Caldwell, Goff & Stanley, 1992; Goff, Verespej & Smith, 1999). Emulsifiers in ice cream promote this process by lowering the fat/water interfacial tension in the mix more than mix proteins,

making their adsorption more favourable during cold aging and resulting in protein displacement from the fat globule surface. This displacement reduces the stability of the fat globule to partial coalescence that occurs during the whipping and freezing process, due to a reduction in protein steric stabilization (Goff, Liboff, Jordan & Kinsella, 1987; Goff & Jordan, 1989; Barfod, Krog, Larsen & Buchheim, 1991; Boode & Walstra, 1993; Gelin, Poyen, Courthaudeon, Le Meste & Lorient, 1994; Gelin et al., 1996a; Gelin et al., 1996b; Pelan, Watts, Campbell & Lips, 1997). Fat destabilization can also be affected by process parameters, particularly the shear induced by the dasher, the overrun content, and the extent of ice formation (Goff & Jordan, 1989; Kokubo, Sakurai, Hakamata, Tomita & Yishida, 1996; Kokubo, Sakurai, Iwaki, Tomita & Yishida, 1998). The phenomenon of fat destabilization in ice cream has recently been reviewed (Goff, 1997).

Due to the importance of the fat destabilization phenomena in ice cream, it would be desirable to predict the extent of fat destabilization for a particular freezing process from measurement of colloidal properties in mix. This would allow ingredient suppliers and manufacturers to assess the impact of formulation changes without

* 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.

extensive pilot plant work. Since the effect of emulsifiers on fat destabilization has been described (Goff & Jordan, 1989; Barford et al., 1991; Pelan et al., 1997), and the effect of emulsifiers on protein displacement from the interface of fat droplets in emulsions is also well known (Goff et al., 1987; Gelin, Poyen, Courthadon, Le Meste, & Lorient, 1994, 1996a), it seems plausible that measurements of adsorbed protein in mix should allow for the prediction of fat destabilization that will subsequently occur during the freezing of that mix under constant processing conditions.

The objectives of this work were to study the effect of emulsifiers on the structure of fat in ice cream mix and ice cream: its subsequent meltdown characteristics; and to establish correlation between colloidal ice cream mix properties and properties of ice cream. A range of emulsifier types and concentrations was used to produce a series of ice cream mixes. The amount of protein adsorbed to the surface of fat globules and fat particle size were measured. After freezing, fat agglomeration index, solvent-extractable fat, fat agglomerate size, microstructure in the frozen state, melting rate and component analyses (protein and fat) of the melt were measured and correlated to mix property measurements.

2. Materials and methods

2.1. Mix and ice cream preparation

Six different ice cream recipes were utilized. All mixes contained 10% milk fat (anhydrous milk fat, Gay Lea, Guelph, Canada), 10% milk solids-not-fat (low-heat 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% alpha 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 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, homogenized at 172/34 bar (APV Gaulin single piston two stage homogenizer, Everett, MA), cooled and immediately stored at 4°C in an ice water bath and aged for one day at 4°C. Batches of ice cream mix (2 L) were frozen in a 4 L batch freezer (Taylor Freezer B733-32, Rockton, IL). During the freezing process the temperature of the ice cream in the freezer was measured continuously by means of a thermocouple integrated in the freezer barrel door. The ice cream samples were drawn 30 s after the indicated temperature reached

– 5.8°C. Immediately after the first sample had been drawn, overrun was measured. Ice cream was hardened and stored at – 27.5°C. Mix and ice cream processing as well as all analyses were carried out in triplicate.

2.2. Analyses

2.2.1. Fat particle size analysis

Particle size distributions of the mix (4°C) and melted ice creams after hardening (4°C for 3–4 h) were measured by integrated laser light scattering, using a Mastersizer X (manufacturer's presentation code 0303, specific for refractive index of milkfat, Malvern Instruments Ltd., Malvern, Worcs., UK). The dilution of the emulsion in the sample chamber was approximately 1 : 1000 with distilled water at room temperature. Mean particle size diameter $d_{4,3}$ (the volume-surface-weighted diameter), specific surface area ($\text{m}^2 \text{mL}^{-1}$), and the cumulative percentage of the particles at 3.0 μm were recorded. This latter value was taken as a measure of the formation of a second peak of fat globule aggregates, since no globules > 3.0 μm existed in the mix.

2.2.2. Electrophoresis

The adsorption of protein on the fat globules of ice cream mix was measured by SDS-PAGE electrophoresis. To obtain fat globules devoid of serum protein, the emulsions were separated by ultracentrifugation (Beckman, preparative ultracentrifuge L8-70M, rotor TI-70) at 15,000 g for 60 min at 20°C. Ice cream mix was warmed to 20°C just prior to centrifugation. These conditions were chosen based on the research of Segall and Goff (1999). Fat crystallization can cause coalescence during centrifugation, thus biasing the results. At 20°C, original fat globule size distributions can be recovered after resuspension of the fat globules, whereas at 4°C they cannot. Sample preparation for SDS-PAGE was carried out as described by Hunt and Dalgleish (1994). Each sample (1 μL) was loaded into a 20% homogeneous "Phastgel" (Pharmacia Biotech), and run in a rapid electrophoresis system (Phastsystem, Pharmacia Biotech) at 15°C. The gels were stained with Coomassie blue (PhastGel Blue R, Pharmacia Biotech) and destained with a solution of distilled water/acetic acid (10%)/methanol (30%) at a concentration of 6 : 1 : 3 for about 1 h. Subsequently the gels were dried at room temperature. After separation, scanning densitometry was performed with a scanner (Sharp JX-330, Sharp Corporation, Japan). The scanned images were evaluated with "Image Master" software (Pharmacia Biotech, Canada). The intensity, the size and the distance between the bands are expressed in a peak diagram. The area below the peak (raw volume) was used for quantification. Reference samples of the ice-cream mix (emulsion) were compared to the cream phase. Adsorbed protein (mg m^{-2}) was calculated from specific surface area data.

2.2.3. Turbidity

To evaluate the degree of fat destabilization by spectroturbidity (Goff & Jordan, 1989), ice cream samples were thawed and an aliquot of 40 mL was taken for analysis. Mix and ice cream samples (3.00 g) were diluted 1 : 500 in two steps with filtered water (MiliQ, Millipore Canada, Mississauga, ON) and absorbance was measured by a spectrophotometer (Shimadzu UV-1201, Kyoto, Japan) at 540 nm against MiliQ water as a blank. Fat agglomeration index was calculated as (absorbance in mix – absorbance in ice cream)/absorbance in mix $\times 100\%$.

2.2.4. Scanning electron microscopy (SEM)

For sample preparation, aluminum tubes (inner diameter 1.16 mm, length 30 mm) were used, which were previously marked for easy breakage. The aluminum tube was pushed twice into the ice cream approximately 1–2 cm below the surface. It was then dropped immediately into liquid nitrogen (-196°C), where it was stored until preparation for microscopy. The sample holder for cryo-SEM consisted of a copper base with vertical holes for tube insertion. Two specimens were placed in the holder while immersed in liquid-nitrogen slush. The holder was then transferred in a vacuum (1×10^{-2} Torr) to the preparation chamber (EMscope SP2000A Sputter-Cryo Cryogenic Preparation System, EMscope Ltd., Kent, England) using the transfer device. The specimens were fractured by breaking the tube at the predefined position using the blade in the preparation chamber and then transferred to the cold stage of the microscope (Hitachi S-570 SEM, Hitachi Ltd., Tokyo, Japan) for sublimation (15 min). After etching, the holders were transferred back to the preparation chamber for gold-sputter coating (2.5 min). Specimens were viewed at 10 kV accelerating voltage with an objective lens aperture of 50 μm .

2.2.5. Melting test

All the samples were stored before melting at -27.5°C . Samples for the meltdown test (260 mL) were removed from the containers and weighed on a scale. The samples were placed on a 10 mesh grid (= 10 holes per 2.54 cm), wire thickness 0.9 mm, 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 regularly. The portion of the ice cream that remained above the screen (denoted below as “remaining portion”) after 2 h was quantitatively removed, weighed and both fractions were analyzed for total fat and protein.

2.2.6. Fat and protein analyses

The original mix and fractions (dripped and remaining portion) from the melting test were analyzed for total protein by Kjeldahl analysis and fat by Mojonnier analysis.

2.2.7. Solvent-extractable fat

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 10 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 3 min, 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 rpm/30 s) 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.

2.2.8. Statistical analyses

Each of the response variables from the three replicates of the six mixes (adsorbed protein, $d_{4,3}$ mix) was correlated with each of the response variables of the six ice creams (fat agglomeration index, solvent-extractable fat, $d_{4,3}$ ice cream, cumulative fat % $> 3 \mu\text{m}$, melting rate, % fat and % protein in remaining portion and dripped portion of the meltdown test) by linear and non-linear regression (SAS Inc., Cary, NC). Correlation coefficients (r^2) were determined from the regression line. Response variables from the six ice creams were also correlated with each other by linear and non-linear regression.

3. Results and discussion

3.1. Effect of emulsifier on mix and ice cream parameters

Emulsifier addition had a significant influence on ice cream mix and ice cream colloidal parameters (Table 1). There was no significant difference seen in the extent of homogenization ($d_{4,3}$ mix values), suggesting that homogenization pressure was the dominating factor in emulsion formation, and there was sufficient surfactant present from either protein or emulsifier to produce satisfactory emulsion size distribution. Gelin et al. (1996a) showed a reduction in mean diameter ($d_{3,2}$) in ice cream mix with increasing mdg, but no further reduction from the use of ps80. Thus, our results confirm their findings with respect to the action of ps80 and suggest that fat globule size in mix is not predictive of subsequent fat destabilization during ice cream freezing. However, as the level of emulsification increased, from increasing

Table 1

The effect of mix emulsifier content on colloidal properties of ice cream mix and ice cream. Standard deviations are shown below each mean value ($n = 3$)

Emulsifier content						
		0.075	0.15	0.15	0.15	0.15
Mono- and di-glycerides	—	0.075	0.15	0.15	0.15	0.15
Polysorbate 80	—	—	—	0.02	0.04	0.06
Fat globule size ($d_{4,3}$, μm) mix	1.3	1.2	1.1	1.0	1.0	1.1
	0.2	0.2	0.2	0	0.1	0
Adsorbed protein (mg m^{-2})	11.4	8.8	9.0	7.5	6.5	6.3
	1.6	0.8	0.6	0.3	1.3	1.6
Fat agglomeration index (%)	4.7	3.3	9.7	36.4	58.0	68.4
	0	0.3	3.4	6.8	11.6	10.1
Solvent extractable fat (%)	22.2	2.1	11.2	45.7	71.9	75.2
	0	0.1	9.8	0.9	6.4	6.9
Fat globule/aggregate size ($d_{4,3}$, μm) Ice cream	2.4	2.4	5.8	13.8	20.5	22.0
	0.2	0.1	2.8	3.6	5.4	9.5
Fat globule/aggregate > 3.0 μm (%)	5.3	5.2	29.0	47.3	62.1	68.3
	0.8	0	12.3	4.6	8.5	13.9

concentration of mdg and further addition of increasing levels of ps80, the adsorbed protein content in the mix decreased significantly (Table 1). Protein displacement by surfactants has been demonstrated in the past (Goff et al., 1987; Barfod et al., 1991; Gelin et al., 1994, 1996a; Pelan et al., 1997). The results reported here indicate that there is an increasing displacement with increasing emulsification levels, and thus proposed mechanisms of emulsifier action based on reduction of steric stabilization of fat globules from reduced protein load seem accurate.

All the ice cream parameters, i.e., fat agglomeration index, solvent-extractable fat, volume–surface-weighted mean diameter of fat aggregates in the ice cream ($d_{4,3}$), and % of the size distribution of fat globule/aggregates > 3.0 μm , increased significantly with increasing levels of emulsification (Table 1). Fat globule/aggregate size distributions from ice creams showing significant levels of fat destabilization became bimodal and indicated the formation of a second peak of aggregates along with the original peak of fat globules from the mix. The maximum cumulation of particles in the second peak was found at a diameter of $30 \pm 4 \mu\text{m}$. The size of agglomerates seemed to remain constant, perhaps due to limited growth of fat aggregates by the presence of air and ice within the structure. However, more and more agglomerates were formed when the emulsification level increased. Barfod et al. (1991), investigating the influence of emulsifiers on the size of fat agglomerates as measured in thawed ice cream, showed that in the absence of emulsifiers, no stable agglomerates could be detected ($d_{4,3} = 1.3 \mu\text{m}$), while in the presence of mdg ($d_{4,3} = 6.6 \mu\text{m}$) and glycerol monooleate ($d_{4,3} = 24.7 \mu\text{m}$), very stable agglomerates were formed. Our results match these findings very well. The effect of increased emulsification level on fat and air structures in the ice cream was examined using cryo-SEM. As emul-

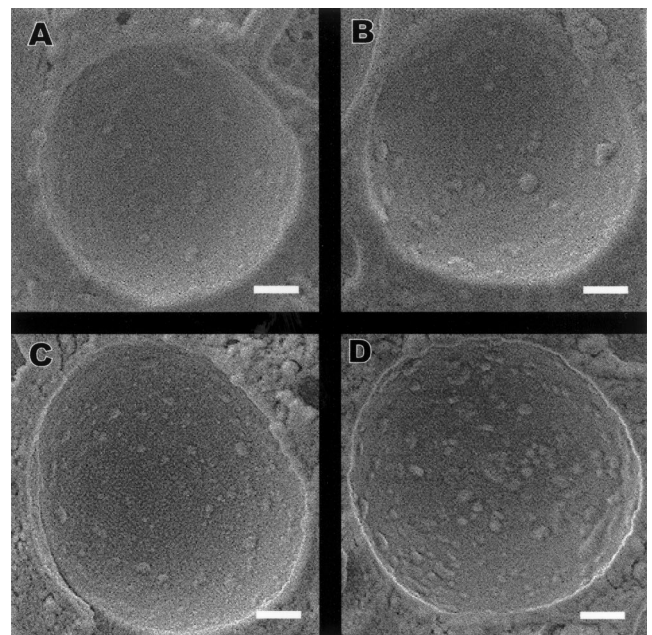


Fig. 1. Cryo-SEM images from sections of air bubbles in frozen ice cream, showing increased adsorption of fat to the air interface with increased emulsification level. 1: No emulsifier; 2: 0.15% mono- and di-glycerides; 3: 0.15% mono- and di-glycerides plus 0.02% polysorbate 80; 4: 0.15% mono- and di-glycerides plus 0.06% polysorbate 80. Bar = 2 μm in all images.

sification increased, more fat globules could be seen at the air interface and these globules seemed to penetrate further into the air bubble with increased ps80 concentration (Fig. 1). Similar images have been presented recently by Goff et al. (1999). This behaviour of fat globules could be expected based on a reduction in protein load and an increased hydrophobicity of the fat globule surface.

As expected, melting rates as a function of level of emulsification decreased (Table 2). However, ice cream samples seemed to be grouped into two populations, those with high melting rates and those with low melting rates. The addition of low levels of ps80 caused a sudden and dramatic decrease in melting rate. In addition to melting rate, shape retention of the “remaining portion” (the portion which stayed above the screen), assessed qualitatively, also showed important differences (Fig. 2). While additions of higher levels of ps80 caused little further effect on the melting rate, noticeable differences in shape retention could be seen. The remaining portion during the meltdown test was able to hold its shape for several hours with little structural collapse after ice had completely melted. This would suggest that appropriate analysis of melting includes a combination of the rate of drip and the shape factor resulting from the extent of fat network formation. The importance of the fat network to shape retention is further substantiated by examining fat particle size analysis in the remaining and dripped fractions. Fat agglomerate size ($d_{4,3}$) in the remaining fraction of the meltdown test increased significantly

Table 2

The effect of mix emulsifier content on ice cream meltdown analyses. Fat and protein concentrations are shown in the portion remaining above the screen and in the portion that had dripped through the screen after 2 h. Standard deviations are shown below each mean value ($n = 3$)

Emulsifier content							
Mono- and di-glycerides	—	0.075	0.15	0.15	0.15	0.15	
Polysorbate 80	—	—	—	0.02	0.04	0.06	
Melting rate (% min ⁻¹)	0.9	1.0	0.8	0.2	0.2	0.1	
	0	0	0.3	0	0	0	
Fat (%)							
Remaining	10.1	10.1	10.9	11.8	11.8	12.0	
	0.1	0.1	0.8	0.2	0.4	0.1	
Drip	10.2	10.1	8.6	2.0	0.6	0.3	
	0.1	0.1	2.9	0.9	0.1	0	
Protein (%)							
Remaining	3.8	3.8	4.0	4.3	4.2	4.2	
	0.1	0.1	0.2	0.1	0.1	0.1	
Drip	3.8	3.8	3.5	1.8	1.7	2.0	
	0.1	0.1	0.7	0.2	0.2	0.1	
Fat globule size ($d_{4,3}$, μm)							
Remaining	2.4	2.4	3.6	18.2	28.0	34.8	
	0.2	0.2	0.2	2.1	3.4	1.6	
Drip	2.4	2.4	3.2	2.1	1.3	2.0	
	0.2	0.2	0.7	0.1	0.1	0.1	

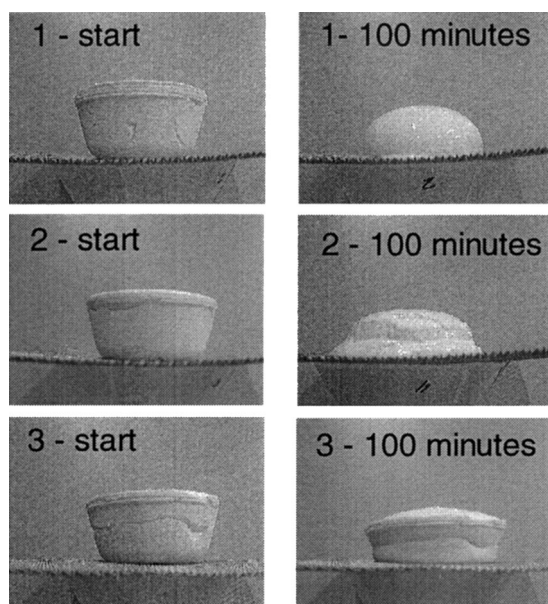


Fig. 2. Shape retention in the portion above the screen (remaining portion) during the meltdown test at the beginning of the experiment and after 100 min. 1: No emulsifier; 2: 0.15% mono- and di-glycerides plus 0.02% polysorbate 80; 3: 0.15% mono- and di-glycerides plus 0.06% polysorbate 80.

(Table 2), and analysis of fat particle size in the dripped fraction revealed that the mean fat globule size for all the mixes was monomodal. Hence, all the agglomerated fat structure was present in the remaining portion and could not leave the network system. This has been suggested previously by Tharp et al. (1998).

Ice cream samples that contained ps80 melted very differently than those containing only mdg, and these differences were not fully explained by the adsorbed protein measurements. It appears that the action of ps80 in ice cream may be quite different than that of mdg. No measurements were made in this study of air bubble size distribution or composition of the air interface, and it is possible that effects of ps80 at the air interface contribute to differences seen in meltdown shape retention. In addition, as seen in Fig. 1 and reported elsewhere (Goff et al., 1999), increasing fat destabilization manifests itself as more fat globules at the air interface, and partially coalesced fat structures in the bulk phase. As ice crystals melt and ice cream structure collapses during ice cream melt down, these fat structures would be expected to hold more of the original shape during and after ice melting. Quantitative analysis of fat and protein contents in the remaining and dripped portions from the meltdown test produced significantly different results from the two portions when ps80 was present (Table 2). Concentrations of fat and protein in the remaining portion increased slightly, whereas fat and protein in the dripped portions decreased more dramatically. This can be explained based on the agglomerate size and structure; as fat network formation increased, the serum carried reduced levels of discrete fat globules as it drained out of the fat network and the fat network helped to retain the original shape.

3.2. Correlation between mix and ice cream analyses

Correlation between adsorbed protein measurements and all of the conventional analyses applied to describe the level of fat agglomeration in ice cream (fat agglomeration index, solvent-extractable fat, fat aggregate size in ice cream ($d_{4,3}$), and fat globule % > 3.0 μm) are presented in Fig. 3. Although the lowest level of emulsifier used (0.075% mdg) produced significantly lower adsorbed protein values than the non-emulsified mix, the extent of fat agglomeration between these mixes did not differ. This suggests that a certain level of protein depletion must be exceeded in order to significantly weaken the fat globule membrane system and allow for partial coalescence to occur. After a critical level of protein adsorption, fat destabilization ceases and therefore further adsorption has little effect. Hence, correlation was much higher and linear relationships were more obvious when considering only the mixes that contained emulsifier. Due to the lack of variation of $d_{4,3}$ values in the mix, this parameter correlated poorly with measures of fat agglomeration.

The determination of protein load in the mix seems to be an early indication for the potential of fat structuring in ice cream. Protein load is based on all the protein fractions measured by means of electrophoresis. Both the casein fraction and the whey protein fraction seemed to decrease by a factor of 2 in their relative coverage (casein

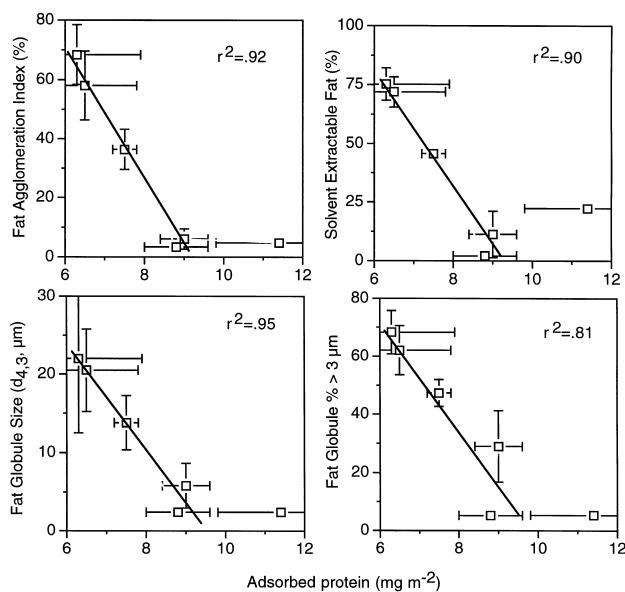


Fig. 3. The relationships between adsorbed protein content of ice cream mix (mg m^{-2} of fat surface area), as obtained by varying the emulsifier content, and indices of fat destabilization in frozen ice cream (fat agglomeration index, solvent-extractable fat, fat globule aggregate size). Error bars are standard deviations ($n = 3$). Linear correlation coefficients (r^2) from the regression line exclude the highest level of adsorbed protein (the non-emulsified mix).

or when protein analyzed in fat fractions compared to casein or when protein in ice cream mix emulsions and expressed in %) as emulsification increased from the lowest to the highest. Hence, the measurement of all fractions as combined in total protein per surface area seems to be a good estimate of the level of ice cream stability. Thus, electrophoresis measurement could be replaced by a simple centrifugation process followed by quantitative protein analysis (e.g., Kjeldahl) in the fat fraction as separated in centrifugation, in order to develop a simplified standard laboratory procedure.

Overrun also increased to a minor extent with an increase of emulsification level (data not shown). While overrun may have had some effect on fat destabilization, it is not possible from this work to isolate the extent of fat agglomeration caused by the reduction of protein load from the one caused by a change in overrun. However, we have observed that conventional continuous freezing at constant preset overrun with the same formulation as the ones reported here produced very similar results in fat agglomeration (data not shown). Hence, it can be concluded that the level of agglomeration obtained in this study was only influenced to a minor extent by a parallel increase in overrun with an increase of emulsification level.

3.3. Correlation of ice cream analyses

Fat agglomeration index (% fat destabilized), solvent extractable fat, and fat agglomerate particle size analysis

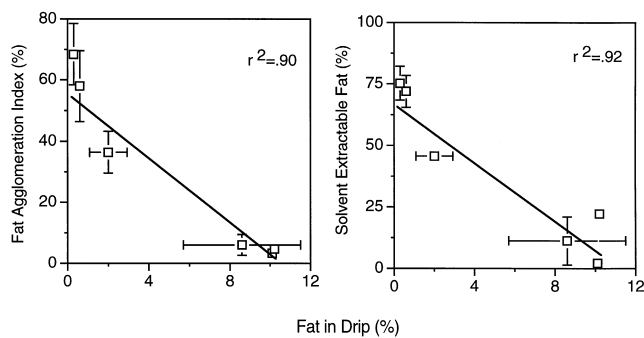


Fig. 4. The relationships between fat content (%) in dripped portion of the meltdown test and indices of fat destabilization in frozen ice cream (Fat agglomeration index, solvent-extractable fat). Error bars are standard deviations ($n = 3$). Linear correlation coefficients (r^2) were calculated from the regression line.

have been used to quantify fat destabilization in ice cream. In this study, all were found to correlate highly. Fat agglomerate size in ice cream ($d_{4,3}$) produced high linear correlation with fat agglomeration index ($r^2 = 0.99$) and with solvent extractable fat ($r^2 = 0.94$). Fat agglomeration index produced a high linear correlation ($r^2 = 0.95$) with solvent-extractable fat. Thus, it appears that while nature of the measurement may differ, the underlying parameter being measured by each technique is the same, and thus any parameter can be taken as an indicator of fat structuring by partial coalescence.

Good correlation was also found between parameters of the meltdown test and indices of fat agglomeration. Melting rate per se did not correlate well with fat destabilization due to the two discrete populations of melting rate seen. As discussed above, a true picture of the effect of fat destabilization on melting only emerges when shape retention is also considered in addition to melting rate. However, high linear correlation to indices of fat agglomeration was found with the analysis of fat in the dripped portion of the melting test (Fig. 4). As well as fat agglomeration index and solvent extractable fat shown in Fig. 4, fat in the dripped portion also correlated well with fat agglomerate size measured by light scattering, as would be predicted based on the high linear correlation of these fat agglomeration indices with each other. Particle size analysis (Table 2) indicated that fat in the dripped portion consisted of discrete fat globules and very little agglomerated fat escaped the fat network in the remaining portion. Contrary to this, discrete fat globules and serum could remain trapped in the remaining portion thus influencing the measurements for the remaining portion. Thus the dripped portion may be a more reliable source of information. The measurement of fat content from the dripped portion of a melting test could be a simple analytical method to characterize the fat structure system in ice cream.

4. Conclusions

A range of six different mix recipes with increasing levels of emulsification had a significant measurable effect on ice cream mix and ice cream properties. Increasing levels of emulsification significantly depleted protein from the fat globule in the mix. The adsorbed protein content in the mix (mg m^{-2} of fat surface area) was a predictive measure of fat destabilization during freezing. As the most important effect for ice cream, an increase of fat destabilization was seen with increasing emulsification, and a subsequent decrease in melting rate and enhanced shape retention during melting could be observed, especially when using ps80 in combination with mdg. Structural analyses indicated enhanced interaction between fat and air as protein adsorption decreased. Importantly, the correlation between adsorbed protein level and fat destabilization indices was found with constant processing conditions. It is known that modifications to process conditions, such as draw temperature or back pressure, can also cause modifications to fat destabilization levels (Goff et al., 1999; Kokubo et al., 1996, 1998), thus correlations established in this research are specific to modifications to emulsification level. Correlation between fat in the dripped portion from the melting test and indices of fat destabilization was high, thus the analysis of total fat from the dripped portion can be taken as a measure of fat destabilization, in addition to parameters such as fat agglomeration index, solvent extractable fat and size of fat agglomerates.

Acknowledgements

Appreciation is expressed to Germantown International Ltd., Botany, NSW, Australia, for providing financial support for the post-doctoral fellowship of Dr. Stephan Bolliger at the University of Guelph, during which this research was conducted.

References

Barfod, N. M., Krog, N., Larsen, G., & Buchheim, W. (1991). Effects of emulsifiers on protein-fat interaction in ice cream mix during aging I: Quantitative analyses. *Fat Science and Technology*, 93, 24–29.

- Berger, K. G. (1997). Ice cream. In S. E. Friberg, & K. Larsson, *Food emulsions* (3rd ed.) (pp. 413–490). New York: Marcel Dekker Inc.
- Boode, K. and Walstra, P. (1993). Partial coalescence in oil-in-water emulsions. 1. Nature of the aggregation. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 81, 121–137.
- Caldwell, K. B., Goff, H. D., & Stanley, D. W. (1992). A low-temperature scanning electron microscopy study of ice cream I. *Techniques and general microstructure*. *Food Structure*, 11, 1–9.
- Campbell, I. J., & Pelan, B. M. C. (1998). The influence of emulsion stability on the properties of ice cream. In W. Buchheim, *Ice cream, International Dairy Federation Special Issue 9803* (pp. 25–36). Belgium: Brussels.
- Gelin, J. -L., Poyen, L., Courthadon, J. -L., Le Meste, M., & Lorient, D. (1994). Structural changes in oil-in-water emulsions during the manufacture of ice cream. *Food Hydrocolloids*, 8, 299–308.
- Gelin, J. -L., Poyen, L., Rizzotti, R., Le Meste, M., Courthadon, J. -L., & Lorient, D. (1996a). Interactions between food components in ice cream. Part 1. *Unfrozen emulsions*. *Food Hydrocolloids*, 10, 385–393.
- Gelin, J. -L., Poyen, L., Rizzotti, R., Dacremont, C., Le Meste, M., & Lorient, D. (1996b). Interactions between food components in ice cream. Part 2. *Structure-texture relationships*. *Journal of Texture Studies*, 27, 199–215.
- Goff, H. D. (1997). Colloidal aspects of ice cream — a review. *International Dairy Journal*, 7, 363–373.
- Goff, H. D., & Jordan, W. K. (1989). Action of emulsifiers in promoting fat destabilization during the manufacture of ice cream. *Journal of Dairy Science*, 72, 18–29.
- Goff, H. D., Liboff, M., Jordan, W. K., & Kinsella, J. E. (1987). The effects of polysorbate 80 on the fat emulsion in ice cream mix: Evidence from transmission electron microscopy studies. *Food Microstructure*, 6, 193–198.
- Goff, H. D., Verespej, E., & Smith, A. K. (1999). A study of fat and air structures in ice cream. *International Dairy Journal*, 9, 817–829.
- Hunt, J. A., & Dalgleish, D. G. (1994). Adsorption behaviour of whey protein isolate and caseinate in soya oil-in-water emulsions. *Food Hydrocolloids*, 8, 175–187.
- Kokubo, S., Sakurai, K., Hakamata, K., Tomita, M., & Yoshida, S. (1996). The effect of manufacturing conditions on the de-emulsification of fat globules in ice cream. *Milchwissenschaft*, 51, 262–265.
- Kokubo, S., Sakurai, K., Iwaki, S., Tomita, M., & Yoshida, S. (1998). Agglomeration of fat globules during the freezing process of ice cream manufacturing. *Milchwissenschaft*, 53, 206–209.
- Pelan, B. M. C., Watts, K. M., Campbell, I. J., & Lips, A. (1997). The stability of aerated milk protein emulsions in the presence of small molecule surfactants. *Journal of Dairy Science*, 80, 2631–2638.
- Segall, K. I., & Goff, H. D. (1999). Determination of protein surface concentration for emulsions containing a partially crystalline dispersed phase. *Food Hydrocolloids*, 13, 291–297.
- Tharp, B. W., Forrest, B., Swan, C., Dunning, L., & Hilmoie, M. (1998). Basic factors affecting ice cream meltdown. In W. Buchheim, *Ice cream, International Dairy Federation Special Issue 9803* (pp. 54–64). Belgium: Brussels.