The influence of emulsifiers on the heat shock stability of ice cream
ABSTRACT
The influence of emulsifiers (mono-diglycerides) on the heat shock stability of ice cream was studied in samples with varying levels of overrun, i.e. 60%, 100% and 140%. Investigations were made to evaluate the size distribution of air bubbles and ice crystals, melting resistance, solvent extractable fat and sensory properties in fresh ice cream samples and samples subject to heat shock. Emulsifiers were seen to increase heat shock stability, melting resistance and air bubble stability and give a finer distribution of air bubbles. Unsaturated emulsifiers provided the best melting resistance. High air content protected ice cream from ice crystal growth when emulsifiers were present. Low air content produced inferior melting resistance.

INTRODUCTION
Good quality ice cream is generally assumed to contain small air cells and ice crystals, resulting in increased creaminess and reduced iciness (1).

The air bubbles in the ice cream foam structure are thermodynamically unstable and may be destabilised by disproportionation, drainage and coalescence (2). Air bubble coalescence is an important mechanism for quality deterioration in ice cream. Foam stability may be improved by the adsorption of partially coalesced fat droplets to air bubbles (3, 4, 5) or by raising the level of viscosity by “hardening” the product (1, 5).

Ice crystal size is a critical attribute governing the texture of ice cream and other frozen desserts. Ice crystals larger than 55mm (X50,0) result in a coarse and icy texture if present in sufficient number (6, 7).

A coarse ice cream structure can result from heat shock during frozen storage or temperature fluctuations during distribution, product mishandling or processing at non-optimal conditions. This can cause large ice crystals and air bubbles to form. Further deterioration can be identified by air channel formation and shrinkage (8).

This paper deals mainly with the influence of air cell structure/air phase volume (% overrun) on ice crystal growth in ice cream with 60%, 100% and 140% overrun. Emulsifiers such as mono-diglycerides and hydrocolloids can stabilise the air structure in ice cream and slow the growth of ice crystals. For this reason, the effect of saturated and unsaturated monoglycerides on heat shock stability was evaluated. This included a study of air cells and ice crystals in fresh ice cream samples and samples subject to heat shock.

The effect of heat shock treatment on the melting resistance and sensory properties of ice cream is also reported, with a particular focus on the difference between saturated and unsaturated monoglycerides.

MATERIAL AND METHODS
Formulations
The recipes used for the trials is shown in table 1 below.

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<td>8.00</td>
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<td>8.60</td>
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<td>-</td>
</tr>
<tr>
<td>Emulsifier no. 2</td>
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<td>-</td>
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<td>Stabiliser blend</td>
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<td>0.18</td>
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<td>Vanilla Flavouring 3000</td>
<td>0.30</td>
<td>0.30</td>
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<td>Colouring</td>
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<td>0.10</td>
<td>0.10</td>
</tr>
<tr>
<td>Total</td>
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<td>100.00</td>
<td>100.00</td>
</tr>
<tr>
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<td>8.10</td>
<td>8.10</td>
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<tr>
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<tr>
<td>Total dry matter</td>
<td>35.80</td>
<td>35.80</td>
<td>35.45</td>
</tr>
</tbody>
</table>

1 Danisco A/S, Brabrand, Denmark, 2 Deutsches Institut für Lebensmitteltechnik, Quakenbrück, Germany.

Table 1. Recipes for the ice cream samples.
Ingredients

- Butter oil concentrate from Arla Foods
- Skimmed milk powder (MILEX® 240) from Arla Foods
- Standard whey powder (Domovictus 100) from Domo Food Ingredients
- Pure crystalline saccharose from Danisco
- Glucose syrup powder, 32 DE (C-PUR 01934) from CERESTAR
- Vanilla Flavouring 3000 from Danisco
- Colourings (water-soluble Annatto A-320-WS) from Chr. Hansen A/S

Processing procedure

The skimmed milk powder, whey powder and a dry blend of sugar, glucose syrup solids and emulsifier/stabiliser blend were dissolved in the water and heated to 50°C; flavour and colour were added and the mix rehydrated for 30 minutes. The fat was melted and mixed with the water phase while being heated to 85°C/30 sec. and then homogenised on a homog- eniser (Lab 1250, APV Rannie) with a LW head at 78°C/180 bar. Subsequently, the emulsion was cooled to 5°C, aged overnight (approximately 24 hours), and frozen and whipped into ice cream with a constant overrun of 60%, 100% and 140% (for sample no. 3, it was only possible to achieve a maximum overrun of 100%) and an outlet temperature of –5.0°C using a Technohoy MF50 continuous ice cream freezer (WCB Ice Cream, Denmark). The ice cream was filled into 0.25 litre cartons and hardened in a hardening tunnel at –35°C for 24 hours. The temperature of the ice cream was raised to and maintained at –18°C for 24 hours before being analysed.

Half of the different samples were subject to heat shock treatment. This involved tempering and storing the products in a freezer at –18°C. After tempering, the products were placed in a heat shock freezer where the temperature varied between –20°C and –5°C every 6 hours. The products were kept in this freezer for 7 days. Both ice cream samples stored at a constant temperature and those subject to heat shock were analysed.

Ice cream analysis

The following analyses were performed on the ice cream samples:

Sensory analysis

The ice cream was evaluated by six trained people from the ice cream group at Danisco, Denmark.

Meltdown determination

The melting rate (drip rate) was measured using the method described in technical memorandum no. 2520 from Danisco (9).

A rectangular piece of ice cream (125cc, dimension: approx. 100mm x 50mm x 25mm), which had been stored at –18°C for at least 24 hours, was weighed and placed on a grid in a room with a constant temperature of 22°C+/–1°C. The grid was placed above a 500ml glass beaker placed on an analytical balance. The analytical balance was linked to a computer which registered the weight of the ice cream once every 2 minutes and calculated the amount of melted ice cream as a function of time. After 2 hours a graph of melting behaviour could be drawn.

Solvent extractable fat (SEF)

The amount of de-emulsified fat was expressed as solvent extractable fat and calculated according to the method described in technical memorandum no. 2517 from Danisco (10).

A 25g sample of frozen ice cream was weighed directly into a number of 100ml cylinder glasses. The ice cream samples were thawed for approximately 2 hours and 30ml heptane filled into each cylinder glass. The lid was carefully closed and the cylinder glasses placed on a rotator.
Rotation: 15 times at 30rpm, followed by 2 minutes’ rest to avoid mix on the surface, then rotation 10 times. The samples were left standing for approximately 60 minutes for separation.

The heptane phase is levered into a ml conical flask which had been dried together with a pumice for 1 hour in an incubator at 105°C and then weighed. The heptane phase was evaporated in a sand bath. This extraction process was repeated twice more, excluding the last 10 rotations. After the final evaporation, the conical flasks were dried in the incubator at 105°C for 4 hours.

Calculation:
Per cent solvent extractable fat = 
gram solvent extractable fat x 100 x 100
25 x F

F = Per cent fat in ice cream sample

Ice crystal and air bubble size distribution – SEM
The structure of the samples was analysed by scanning electron microscopy (SEM) using the cryo-break technique. Afterwards the surface was etched by sublimation of some surface water for a few seconds. The samples were prepared at −25°C in a freezing room. Small pieces of the centre of the samples were taken using a scalpel. These pieces were fixed in the sample holder which was then placed in a closed chamber to transfer the sample to the preparation unit of the microscope. The sample on the sample holder was quickly inserted in the preparation unit and cooled to about −180°C within a few seconds by liquid nitrogen under vacuum. This very rapid temperature decrease is necessary to guarantee no further growth of the existing ice crystals and that no visible new ice crystals are formed. At this temperature the sample was broken using a knife and a smooth surface obtained. Afterwards the surface was etched for a few seconds for better visualisation of the ice crystals. The sample was then sputtered with a thin gold layer to enable electron microscopy.

After this preparation, the sample was transferred directly to the microscope for structure analysis. The temperature was kept at −160 to −180°C using liquid nitrogen. A magnification of 100x and 200x was used. On the photographs the magnification is given by a bar, which represents a length of 100mm. With a few samples a higher magnification of 3,000x was used to study the air serum interface in detail. On these photographs the magnification is expressed by a bar representing a length of 10mm.

Ice crystal and air bubble size distribution were calculated by image analysis (NIH Image 1.6) of the photos. Particle segmentation was carried out manually. Clustered ice crystals separated by the continuous phase were calculated as single crystals. Clustered ice crystals without a visible separation were analysed as one crystal. After segmentation of areas, which refer to ice crystals or air bubbles, the particle size distribution of the ice crystals and air bubbles was computed. The sizes were calculated as diameters of a sphere with the same projection area.

Further characteristic particle diameters are:

\[ x_{10.0}, x_{10.3} \]

10% of the total number or volume of particles have smaller diameters and 90% larger.

\[ x_{90.0}, x_{90.3} \]

90% of the total number or volume of the dispersed particles have smaller diameters and 10% larger.

\[ \text{Span}, width \text{ of the distribution} \frac{x_{90.1} - x_{10.1}}{x_{50.3}} \]

RESULTS AND DISCUSSION
Sensory evaluation
Constant temperature storage
The samples made with unsaturated MG (sample 2) were the creamiest and had the highest body and warmest eating properties. The samples made without emulsifiers were the least creamy and had the least body and coldest eating properties. The creaminess and warm eating properties of the ice cream increased with a higher overrun.

After heat shock
The quality of all the samples decreased following heat shock treatment. The samples without MG showed signs of shrinkage and had the least body and coldest eating properties and were least creamy. The samples made with 140% overrun were fluffy and large air bubbles were visible on the surface. The quality of the ice cream made with saturated and unsaturated MG was comparable.

Solvent extractable fat
Solvent extractable fat (SEF) was used to quantify the amount of de-emulsified fat in the ice cream samples. Increased SEF figures usually indicate higher creaminess and improved melting resistance. For normal bulk ice cream (with 100% overrun) a SEF figure of around 10 is normal. Both increased mechanical treatment of ice cream (lower drawing temperature from the freezer) and a higher degree of emulsion destabilisation (use of emuls-
fiers such as mono- and diglycerides) produce higher SEF figures.

Table 3 shows the influence of overrun and emulsifiers on SEF.

<table>
<thead>
<tr>
<th>OVERRUN</th>
<th>60%</th>
<th>100%</th>
<th>140%</th>
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<tbody>
<tr>
<td>Sample 1 with saturated MG</td>
<td>6.5%</td>
<td>9.0%</td>
<td>10.7%</td>
</tr>
<tr>
<td>Sample 2 with unsaturated MG</td>
<td>15.7%</td>
<td>19.5%</td>
<td>22.0%</td>
</tr>
<tr>
<td>Sample 3 without MG</td>
<td>0.7%</td>
<td>0.5%</td>
<td>–</td>
</tr>
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</table>

Table 3. Solvent extractable fat measured in the ice cream samples.

Table 3 shows that unsaturated MG produced a much higher level of de-emulsified fat than saturated MG. Ice cream without MG produced practically no extractable fat, which means very little fat agglomeration.

**Meltdown determination**

**Constant storage temperature**

Figures 2A, B and C show the melting behaviour of ice cream with different levels of overrun prior to heat shock. In ice cream with 60% overrun (figure 2A), melting resistance improved only slightly using saturated MG, compared to ice cream without MG. Further improvement was achieved using unsaturated MG.

In ice cream with 100% overrun (figure 2B), melting resistance improved considerably using saturated MG, compared to ice cream without MG. Further improvement was achieved using unsaturated MG.

In ice cream with 140% overrun (figure 2C), melting resistance was improved when unsaturated MG was used, compared to ice cream with saturated MG.

Melting resistance improved when overrun was increased from 60% to 100% and when saturated and unsaturated MG were used. But there was no improvement in the melting resistance of ice cream without MG. Increasing the overrun from 100% to 140% also improved the melting resistance of ice cream made with saturated and unsaturated MG.

**After heat shock**

Figures 3A, B and C show the melting behaviour of ice cream with various degrees of overrun after heat shock.

The melting resistance of all the samples subject to heat shock was inferior compared to the other samples. The samples with unsaturated MG were least affected by heat shock treatment.

**Mechanism behind improved melting resistance with unsaturated MG**

In ice cream production unsaturated MGs result in drier extrusion and slower meltdown properties than saturated MGs. Unsaturated MGs have a more destabilising effect on the emulsion of ice cream mix, as can be seen by the higher SEF figures in the analysis. Further differences between unsaturated and saturated MGs have recently been studied in great detail (11). This work showed the two MG types differ in their effect.
on the crystallisation of the fat globules in the mix. Saturated MG preserves fat’s globular state while unsaturated MG induces fat crystal growth penetration on the fat globule surface. This phenomenon is shown in figure 4. The protruding fat crystals are much more efficient in stabilising air bubbles than round fat globules. When using harder fats, such as hardened palm kernel oil, a complete transformation of fat globules to fat crystal plates can be observed (11).

Using saturated MG the fat may have insufficient surface area to stabilise the foam on extrusion via a complete fat network. This is what can be seen to the left in figure 5.

Using unsaturated MG there may be a surplus of fat globules/crystals as indicated to the right in figure 5. Calculations indicate the fat surface area may increase up to 10 times when a fat globule is transferred to a fat crystal platelet. Hard fat thus makes it possible to observe air bubbles stabilised by multi-layers of pure fat crystal platelets.

The difference between unsaturated and saturated MGs in terms of functionality is believed to be due to their behaviour in ternary systems containing water, emulsifier and triglyceride oil (11). It is also related to the differences observed in binary systems of emulsifier and water (12).

In short, unsaturated MGs react more with water at 5°C than saturated MGs, resulting in water penetration into the surface layers of the fat globules and leading to protein desorption and crystallisation of super-cooled fat. Water penetration into the oil phase is increased by an increasing dosage of unsaturated emulsifier (13).

**Air bubble and ice crystal size distribution analysis by SEM**

The characteristic particle diameters of the number and volume distributions are summarised in tables 4 and 5.
Effects of overrun on air bubble size distribution

In general, a higher overrun increased the sizes of the larger fraction of air bubbles both before and after heat shock, see figure 6 for results with saturated monoglyceride.

Effects of heat shock on air bubble and ice crystal size distribution

Figure 7 shows the effect of heat shock on the growth of ice crystals and air bubbles in the 3 samples with 100% overrun.

Both ice crystals and air bubbles grew after heat shock, although to a much lesser degree in the samples with emulsifiers.

Air bubble size

The number distributions of the air bubbles were characterised by slightly smaller air bubble diameters in the samples with unsaturated emulsion than those with saturated emulsifiers. In terms of volume distribution the differences were not significant.

After heat shock, there was a change to a bimodal distribution of the air bubbles in samples with unsaturated emulsifier. That means both the number of very small air bubbles (lower X50.0 value) and the diameters of the large air bubbles increased. This is a typical effect of diffusion. In volume distribution, no differences could be detected between the samples with unsaturated and saturated emulsifiers.

In the ice cream samples which contain only a stabiliser blend but no emulsifiers very large air bubbles were seen in number and volume distribution. After heat shock, a strong increase was observed in air bubble size in number and volume distribution over the whole range of particle diameters. This indicates that both diffusion processes and coalescence lead to air bubble growth.

In general, increasing air bubble sizes were observed with increased overrun, both before and after heat shock. However the samples with emulsifiers produced much finer air distribution than the sample without emulsifier. The sample without emulsifiers had very large air bubbles both under constant temperature storage and after heat shock. For comparison purposes, the mean volume diameter X50.3 of the air bubble size distribution in the 3 samples is shown as histograms in figure 8.

Table 5. Characteristic ice crystal diameters.

<table>
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<tr>
<th>SAMPLE CODE</th>
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<th>X90.0</th>
<th>SPAN0</th>
<th>X50.3</th>
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<th>X90.3</th>
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<td>1A</td>
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<td>13.93</td>
<td>47.25</td>
<td>1.19</td>
<td>44.36</td>
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<td>35.04</td>
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<td>1C</td>
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<td>83.48</td>
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<td>96.90</td>
<td>60.31</td>
<td>119.73</td>
<td>0.61</td>
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</table>

The small air bubbles contribute more to number distribution. The extreme difference between samples 1 and 2 with emulsifiers and sample 3 without emulsifiers in stabilising small air bubbles is shown in figure 9.

In figure 10 SEM pictures show the stabilising effect of MGs on small air bubbles.

Ice crystal size

The sample with saturated MG and the lowest overrun (1A) contained the smallest ice crystals and sample 1C with an overrun of 140% the largest when stored under constant conditions. During heat shock, there is a strong growth in ice
Crystal size in the samples with overruns of 60 and 100%. The higher overrun of 140% seems to protect the ice crystals from growth – sample 1C contained the smallest ice crystals.

In samples 2 with unsaturated MG both after storage under constant conditions and after heat shock storage, the sample with the highest overrun shows the smallest ice crystal diameters in number and volume distribution. This also indicates high overrun protects against ice crystal growth.

**Figure 7.** Scanning electron microscopy of ice cream at 100% overrun – effects of heat shock. A: air bubble; C: ice crystal.

**Figure 8.** Mean volume diameter $X_{50.3}$ of air bubble size distribution.

**Figure 9.** Mean number diameter $X_{50.0}$ of the air bubble size distribution.

**Figure 10.** Scanning electron microscopy of ice cream with 100% overrun. A: air bubble; C: ice crystal.
In samples 3 containing only a stabiliser blend but no MG, the number distribution of the ice crystals is in the same range as samples 1 and 2. The volume distribution shows a greater amount of large ice crystals. The heat shock treatment of samples 3 caused strong ice crystal growth, which can be seen both in number and volume distribution.

Sample 3B with an overrun of 100% and very large air bubble diameters contained larger ice crystals than sample 3A with an overrun of 60%, but after heat shock the ice crystals in the sample with 60% overrun grew considerably.

For comparison purposes, the mean volume diameter $x_{50.3}$ of the ice crystal size distribution for samples 1, 2 and 3 are shown as histograms in figure 11.

This study shows that a higher overrun leads to larger air bubble diameters. Heat shock treatment causes air bubble and ice crystal growth.

In the system where the air bubbles were not stabilised by agglomerated fat (sample 3 without MG), the heat shock treatment caused strong air bubble and ice crystal growth, so the critical mean ice crystal diameter of 55mm ($x_{50.3}$) was reached. This created a rough, icy mouthfeel. Single, very large air bubbles can lead to shrinkage in samples subject to further storage.

In samples 1 and 2 with MG, where the air bubbles were stabilised by agglomerated fat, the samples with the highest overrun of 140% showed the smallest ice crystal diameters after heat shock. Thus, high air content seems to protect the ice crystal from growth. Flores & Goff (1999) reported similar results and found that "higher overruns reduced ice crystal sizes, whereas lower overruns had no impact on distributions, because they did not affect the microstructure of the frozen product" (14).

It was difficult to differentiate between the two emulsifier/stabiliser systems studied with regard to effects on ice crystal size distribution. Concerning air bubble growth, a tendency towards a change to bimodal air bubble distribution was seen in samples 2 made with unsaturated MG. In samples 1 made with saturated MG growth over the whole range of air bubble diameters was detected.

**CONCLUSIONS**

Ice cream with unsaturated MG contained more destabilised fat than ice cream with saturated MG. Ice cream without MG had no destabilised fat. Increased melting resistance was found with increasing fat destabilisation (samples with unsaturated MG were better than samples with saturated MG). Fat destabilisation resulted in highly stable small air bubbles.

**REFERENCES**

9. Determination of the melting rate of ice cream, Technical Memorandum no. 2520, Danisco.
10. SEF Figures - solvent extractable fat, Technical Memorandum No. 2517, Danisco.
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