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THE ADSORPTION OF CRYSTALLINE FAT TO THE AIR-WATER INTERFACE OF WHIPPED CREAM

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Abstract

The interfacial structure of air bubbles in normal and defective whipped creams were compared, using freeze fracturing and transmission electron microscopy, in an attempt to understand the underlying mechanism of the observed gross differences in their whipping times and overruns. In normal whipped creams sparsely distributed fat crystals were found to have penetrated some of the bubbles and were lying in the plane of the air/water interface.

In defective whipped creams, large numbers of needle-like crystals had penetrated the air/water interface of every bubble and, as a consequence, reduced numbers of fat globules were found to have adsorbed. There was also morphological evidence that the crystals reached the interface before the milk fat globules during the whipping process. The presence of large masses of free fat in the aqueous phase of whipped cream, on whose surface arrays of very long fat crystals were found, suggested that the needle-like crystals were dislodged by the shear forces generated during whipping and were then free to adsorb to bubbles. The detection of such large amounts of free fat indicated a large scale damage to fat globules during processing with the consequent escape of both crystalline and liquid fat.

Possible mechanisms to account for the low overrun and long whipping times in defective creams are discussed.

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Introduction

The development of a stable structure in whipped cream depends on interactions between fat globules and between globules and air bubbles. The stabilization of air is a two step process involving the selective adsorption of whey proteins to the bubble surface, thus forming a protein air/water interface, followed by the adsorption of fat globules (Brooker et al., 1986). The fat/water interface of the adsorbed globules coalesces with the air/water interface of the bubble in such a way that part of the fat becomes exposed to the air and slightly protrudes into it (Buchheim, 1978; Brooker et al., 1986). In the final product, bubbles are stabilized predominantly by fat globules but remnants of the initial protein interface persist between the globules. For these events to occur, it is essential that the globules should contain a proportion of crystalline fat by conditioning at 4°C for 24h.

Thus, several authors have found that when the air/water interface is viewed *en face* using freeze fracturing techniques in conjunction with scanning electron microscopy (cryo-SEM), the smooth surface of the protein interface and the attached fat globules can be clearly seen (Schmidt and van Hooydonk, 1980; Brooker et al., 1986; Anderson et al., 1987; Anderson and Brooker, 1988).

Since this hybrid air/water interface of protein and fat is so important for bubble stability, changes in interfacial structure may be invoked to explain why some creams produce defective whipped creams with poor stability or low overrun. Previous studies, in which instability in whipping cream was investigated using cryo-SEM, concluded that the aggregation of fat globules, initiated during the commercial process of separation, was instrumental in causing foam collapse but no changes in the air/water interface were observed at this level of resolution (Anderson et al., 1987). In the present study, the structure of the air-water interface of normal whipped cream was compared with that in a series of defective creams that would not whip normally. For this purpose, freeze fracturing techniques in conjunction with transmission electron microscopy were used after preliminary studies with cryo-SEM failed to produce any conclusive results.

Materials and Methods

Whipping cream

Normal whipping cream was obtained by separating bulk tank summer milk from the Institute's Friesian herd. Milk was pasteurized at 72°C for 15s and separated at 45°C using an Alpha Laval laboratory separator. The cream, whose fat content was adjusted from 42% to 38% (w/w), was conditioned by holding at 4°C for 24h; triplicate samples of 4 different creams were then whipped as described below.

Six samples of defective, 2 day old, refrigerated cream of 38% (w/w) fat content were obtained already separated and pasteurized from a number of commercial farm sources between late June and early September. All of the creams were noticeably more viscous than the control samples but quantitative measurements of viscosity were not made. The cream was produced by separating milk to 53-55% (w/w) fat content and adjusting to 38% (w/w) using skimmed milk. Creams were held at 4°C for 24h and then triplicate samples were whipped. All of these creams were reported by the suppliers to be difficult or impossible to whip.

All creams were whipped at 4°C using apparatus based on the design of Mohr and Koenen (1953) as modified by Scurlock (1983). Cream was whipped to maximum stiffness as measured by the load required to turn the blades. The overrun (volume increase) was calculated by comparing the weight of a volume of unwhipped cream with the weight of the same volume of whipped cream in the manner suggested by Rothwell (1964).

Electron Microscopy

Freeze fracturing and replication was performed using a Balzer BAF 400D unit. Small volumes (1-2 mm^3) of whipped cream were placed into normal freeze-fracture gold specimen holders, cryofixed rapidly by plunging into melting propane (-189°C) and then transferred to liquid nitrogen. In the case of the unwhipped creams, flat copper holders of the sort used for complementary replicas were also used. No cryoprotectants were used. Freeze fracturing was performed at -120°C, followed immediately by replication with 2 nm of platinum/carbon at an angle of 45 degrees and 20 nm of pure carbon from electron beam guns. Replicas were cleaned for 1 h in a 5% (w/v) sodium hypochlorite solution followed by washing in several changes of distilled water and acetone. Replicas were mounted on naked copper 300 mesh grids and examined in a Hitachi H-600 transmission electron microscope at an accelerating voltage of 100 kV.

An indication of the number of adsorbed fat globules/µm^2 of the air/water interface of the bubbles in normal and defective creams was obtained by counting the number of globules in a known area of total bubble interface directly from micrographs. Errors in area measurement resulting from slight differences in curvature of bubbles were not corrected in this approximation.

Results

Normal whipped cream

The whipping characteristics of the normal creams are given in Table 1 and show that overrun and whipping times were within acceptable normal limits.

Table 1. Comparison of the whipping properties of normal and defective creams containing 38% (w/w) fat. Mean values ± S.D..

	Overrun/%	Whipping time/s	Stiffness/g
		Normal cream	
1	92 ± 5.1	107 ± 5.6	78.7 ± 9.3
2	102 ± 7.3	95 ± 7.3	81.2 ± 5.8
3	98 ± 6.1	110 ± 9.9	85.6 ± 8.1
4	110 ± 8.9	92 ± 6.8	88.2 ± 9.7
		Defective cream	
1	18 ± 3.7	230 ± 14.3	64.2 ± 4.4
2	23 ± 2.9	174 ± 6.6	57.3 ± 5.7
3	35 ± 1.8	142 ± 9.5	68.3 ± 6.5
4	18 ± 4.5	167 ± 12.7	41.5 ± 3.9
5	14 ± 1.2	225 ± 11.1	54.8 ± 2.2
6	25 ± 3.7	238 ± 8.8	43.6 ± 3.3

In replicas of the frozen and fractured whipped creams the surface of the air-water interface of the bubbles consisted of areas of relatively smooth whey protein penetrated by numerous fat globules (Fig. 1). Close examination of the globules showed that the characteristic peripheral triglyceride crystals described by Buchheim and Precht (1979) were exposed to the air and that, as expected from a previous study (Brooker et al., 1986), the junction of the

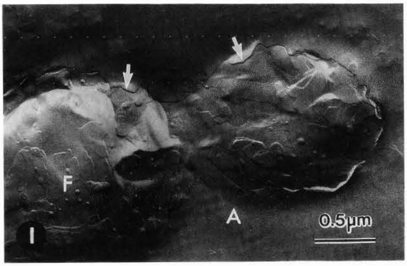
Fig. 1 Part of two adjacent milk fat globules (F) adsorbed to an air bubble from normal whipped cream. The crystalline triglyceride at the surface of the globules can be seen together with the surrounding smooth surface of the protein air/water interface. The junction of the air/water interface with the fat/water interface of the globule is marked by a distinct line around the edge of the globules (arrows). Prepared by freeze fracturing (FF).

Fig. 2 An *en face* view of the concave surface of a bubble in normal whipped cream. A fat crystal (C) is adsorbed to the air/water interface. The small bumps are impressions of casein micelles caused by expansion of the aqueous phase during freezing. FF.

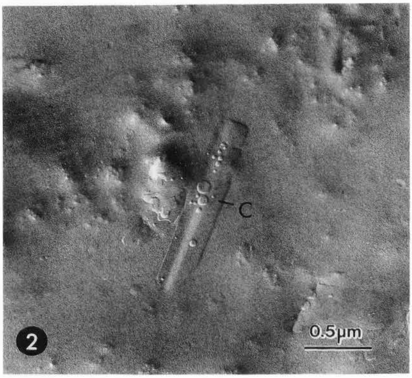
Fig. 3 The concave surface of bubbles from a) normal and b) defective whipped creams at the same magnification. FF.

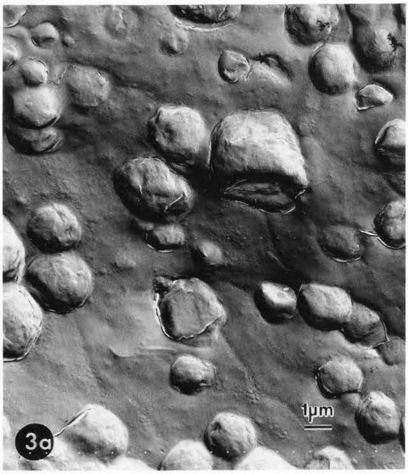
a) This shows many adsorbed fat globules projecting into the air. The relatively smooth surface between the globules is protein air/water interface. b) There are markedly fewer adsorbed globules compared with (a). Numerous needle-like fat crystals (C) project into the air and lie in the plane of the air/water interface. The line across the centre of the micrograph is a tear in the replica.

Fig. 4 Bundles of crystals lying in the plane of the air/water interface of a bubble from defective whipped cream. F= fat globule. FF.


F
A
0.5µm
1


F
0.5µm
4


C
0.5µm
2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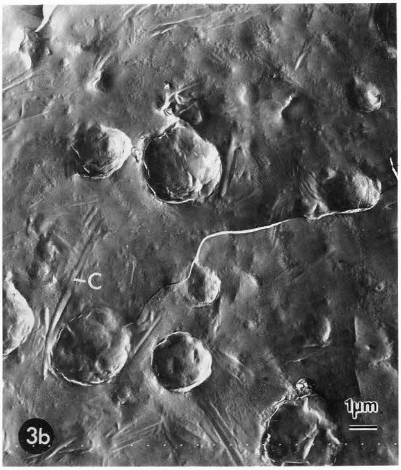
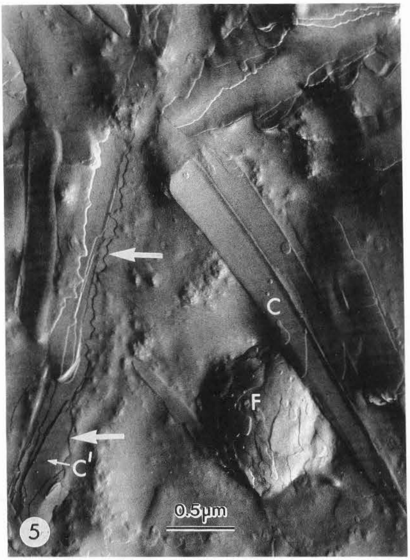
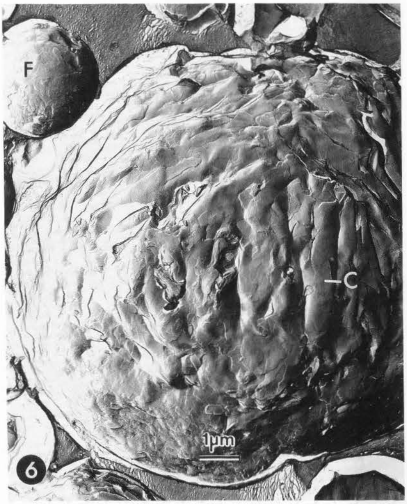
3a


C
1µm
3b





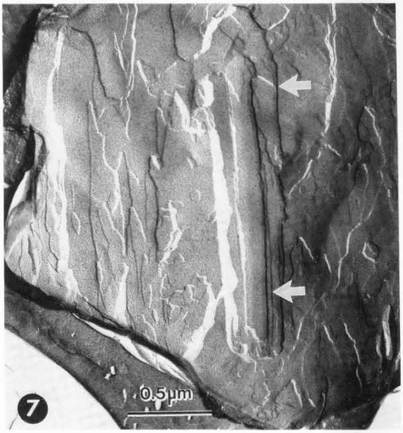


Fig. 5 Concave surface of a bubble in defective whipped cream. A milk fat globule (F) lying behind and partially obscured by an adsorbed lipid crystal (C) indicates that the crystal arrived at the air/water interface before the globule. Note that in some places, breaks in the protein air/water interface can be seen where crystals (C') have adsorbed. In this micrograph the edge of the interface appears as an undulatory line (arrows). FF.

Fig. 6 A large globular mass of free fat from the aqueous phase of defective whipped cream containing large crystal aggregates (C). Note the size of a normal milk fat globule (F). FF.

Fig. 7 The surface of part of a mass of free fat showing an aggregate of fat crystals (arrows). FF.

air/water interface with the fat/water interface was marked by a distinct line at the edge of the globules (arrows, Fig. 1).

In addition, every sample contained a small proportion of air bubbles which, at some point over their surface, possessed 1 or 2 small (1-1.5 µm long) adsorbed fat crystals lying in the plane of the air/water interface Fig. 2). They were similar in size and appearance to crystals previously described by Buchheim et al. (1985) from whipped UHT-treated imitation cream.

In the aqueous phase of both the starting cream and the whipped cream, globules were of normal size and appearance with no evidence of free fat. Free fat is defined here as fat which has escaped from milk fat globules following mechanical damage to the milk fat globule membrane. It can be recognised by electron microscopy because of 1) the absence of the characteristic arrangement of small peripheral triglyceride crystals referred to above and 2) its tendency to form droplets considerably larger than the fat globules .

Defective whipped cream

The whipping properties of the defective creams given in Table 1 show that whipping times were very long and produced exceptionally low overruns. The small numbers of bubbles found in replicas of the frozen and fractured whipped creams were consistent with the low cream overruns. In addition, the number of adsorbed fat globules at the air/water interface was consistently smaller than that found in normal whipped creams (Fig. 3). The mean number of adsorbed fat globules/μm^2 of bubble interface was 0.06 for defective creams and 0.17 for normal creams.

In addition to the reduced number of fat globules, the appearance of the air/water interface was grossly abnormal. Large numbers of very long, and apparently randomly orientated, needle-shaped structures were found protruding into the air and lying in the plane of the air/water interface (Fig. 4). These needles were identified as fat crystals on the basis of their layered structure (i.e. crystal monolayers) and their resemblance to known crystalline lipid described from other food systems (e.g. Buchheim et al., 1985).

Because some of the adsorbed fat globules were partially covered with one or more crystals in en face views of the bubbles, it is evident that during the whipping process many of the crystals adsorbed to the air/water interface before the globular fat (Fig. 5). At the point where the fat crystals penetrated the bubble, the full thickness of the protein air/water interface could be seen. This is particularly easy to see in Fig. 5 where the air/water interface appears as an undulatory layer on the surface of a crystal and has a high contact angle with the latter.

Examination of replicas of the aqueous phases of both the starting cream and the whipped product showed fat globules of normal appearance and frequent globular masses of free fat up to 30 µm in diameter (Fig. 6). At the periphery of these masses, arrays of long crystals similar to those found in the air/water interface of the whipped creams were observed (Fig. 7); the overall appearance of the fat was quite different from that found at the surface of normal milk fat globules.

Discussion

Fat globules are highly susceptible to damage during processing. It is evident from the presence of free fat in the defective creams considered in this study that globular fat had been markedly damaged at one or more of the processing steps in production . Both Rothwell (1966) and Foley et al. (1971) have drawn attention to the rapid increase in free fat when cream is separated above 35 - 40% fat (as was the case with the defective cream) and have underlined the advisability of separating to a value close to the required fat content rather than separating to a higher value and then diluting. These authors also make it clear that even 'normal' whipping cream will always contain some free fat.

The fat crystals observed in this study may arise as a result of crystallization from the liquid fat released from any globules that are damaged whilst still at a high processing temperature. In view of the work of van Boekel and Walstra (1981) on the role of crystals on globule destabilization, it is also possible that they are derived from globules which have been cooled after processing and which therefore release a mixture of crystalline and liquid fat when they are damaged.

It is evident from the normal whipped creams that some fat crystals are available for adsorption to the air/water interface during whipping. These are so few in number that they can have little or no effect on the whipping process or the quality of the final product. However, this observation, together with a similar report from UHT treated imitation cream by Buchheim et al. (1985), suggests that the phemonenon of crystal adsorption to bubbles is extremely common in whipped oil-in-water emulsions.

In the defective cream however, the crystalline fat is a conspicuous component of the air/water interface and probably exerts a marked effect on interfacial behaviour. The large number of fat crystals adsorbed to the air bubbles together with the observation that many crystals adsorb before the fat globules reach the interface, may explain why the number of globules attached to air bubbles is greatly reduced compared with control whipped creams. The successful adsorption of a sufficent number of fat globules to the air/water interface is important for the formation of a network of globular fat and, thereby, for the development of a whipped cream with stiff, dry and stand up properties. The involvement of crystals at the interface helps explain why these desirable properties are not seen to any marked degree in the defective whipped creams.

Fat globule damage, as envisaged above, also leads to the release of liquid fat which is known to have a marked destabilising effect on air bubbles in dairy products. Following adsorption, droplets of liquid fat spread at the air/water interface and there is local thinning of the protein film with subsequent collapse of the bubble (Prins, 1986). This effect may be responsible for the very low overruns and long whipping times seen in the defective whipped creams but it was probably moderated by the bubble stabilizing effect of adsorbing fat crystals of the kind reported by Hoerr (1960) for crystals of shortening in cake batters. It can be envisaged that the net equilibrium position between these destabilizing and stabilizing effects will greatly affect the bulk properties of whipping creams containing lower levels of free fat than those used in the present study .

Because of the similarity of their appearance, the fat crystals at the air/water interface of bubbles almost certainly originate from the crystal arrays seen in the masses of free fat in the aqueous phase. It seems most probable that long crystals become detached from the larger masses of fat by the shear forces generated during whipping and that they are then free to attach to the interface of newly formed air bubbles. Examination of Fig. 5 shows that the crystals are considerably longer than could be accommodated in even the largest of the fat globules and therefore they must have undergone growth in the bodies of free fat during storage at low temperature.

It is clear that the number of fat crystals adsorbed to the air/water interface in whipped cream provides a method of assessing levels of free fat in the whipped product and, thereby, cream quality. So far, attempts to reproduce the phenomenon of crystal adsorption to the air/water interface by the addition of churned butterfat or crystalline butterfat fractions to normal cream prior to whipping, have failed.

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Discussion with Reviewers

E. Dentan: In our experience, replicas obtained with fatty material are difficult to clean, and sodium hypochlorite solution is not always adequate. Did you encounter similar problems ?

Author: The inclusion of acetone, or a mixture of acetone and methanol, in the cleaning procedure usually removes the last traces of fat. The problem with this approach is that replicas have a tendency to fold up in acetone more than when water is used as a solvent.

E. Dentan: Did you really have no etching after the freeze-fracture ? In Fig. 5, it appears that a short etching was performed in order to release the edge of the interface.

Author: The inclusion of an etching step in the procedure for the preparation of replicas was not considered necessary to reveal the structures on the air side of the air/water interface. However there may have been some etching between fracturing the samples and coating.

I do not think that etching would be required to reveal the edge of the interface at the point where it was in contact with the triglyceride crystals.

P. Walstra: Part of my interpretation of your interesting results is as follows. During processing of high-fat cream at conditions where part of the fat is crystalline, considerable partial coalescence may occur, leading to large clumps of fat globules. Subsequent temperature fluctuations may lead to a further fusion of the globules in a clump and to the formation of very large fat crystals. Some of these crystals become situated at the oil-plasma interface. During whipping, the clumps become attached (adsorbed) to the air/plasma, but, due to their large size, the shearing forces are often sufficient to tear them off again. The surface forces holding a globule or clump are proportional to its radius, the shearing forces proportional to its radius squared. Because the surface tensions between crystal and air and that between crystal and oil are about equal, it is quite possible that one or two of the large fat crystals remain at the air/plasma interface when a clump is removed. Moreover, the liquid fat tends to spread over the air/water interface due to capillary forces, but this is counteracted (at least slowed down) by capillary forces in the network of fat crystals in the clump and, due to the coarseness of the fat crystals in a clump, liquid fat can much easier be "pulled out" than is the case in original fat globules, with their much finer crystal network. Would you agree with this extension of your own interpretation ?

Author: I believe that the bodies of fat from which the crystals arise are globules of free fat without an intact fat globule membrane - I do not subscribe to the view that they arise from the fusion of fat globules.

The notion that the bodies of fat attach to the air water interface and then detach leaving crystals behind is an interesting alternative to the one given in the paper.

E.H. Lucassen-Reynders: The author puts the whole blame for the defective nature of the unwhippable creams on the fact that these creams initially had been separated to a higher fat content than the normal creams. I wonder if this factor is sufficient to explain the very marked differences in the amounts of solid fat completely wetted by the water phase. Complete wetting of solid fat by the aqueous phase can occur only if the oil/water interfacial tension is below a critical value. If this is the case for the defective creams, but not for the normal ones, one would suspect differences in concentrations of surface-active material between the two types of creams. Does the author have any information on the interfacial tension of his creams to check this possibility ?

Author: This is an interesting possibility that was not considered in the present work. I have no information on the surface tensions of the creams used, but it seems a useful parameter to measure in future work.