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#### THE EFFECT OF PROCESSING ON SOME MICROSTRUCTURAL CHARACTERISTICS OF FAT SPREADS

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#### Abstract

The processing and the composition of fat spreads determine to a large extent the final product properties such as hardness, spreadability, mouthfeel, emulsion stability and salt release. In establishing the relation between composition, processing and final product properties, microstructural studies play an important role. In this context the influence of some process parameters in the production of shortenings and 80% fat spreads on microstructure and product properties has been investigated. Shear, cooling regime and crystallization conditions influence both the emulsion structure and the fat crystalline matrix. In general, working leads to softer products with a more granular crystalline fat matrix, whereas the water droplet size distribution is influenced in a complicated manner by the conditions of shear. This type of work indicates ways to control and manipulate the microstructure and product properties of fat spreads.

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Key words: Processing, microstructure, shear, cooling regime, deformation, A-unit, C-unit, shortening, margarine, crystalline matrix, crystalline shell.

#### Introduction

Composition and processing are important variables in the manufacture of fat spreads. Much skill is required to find an optimal compromise between fat blend composition, processing and desired product properties (7), such as hardness, spreadability, mouthfeel, emulsion stability and salt release. It is considered that, while on one hand, these functional properties are linked with the product microstructure, on the other hand, composition and processing are the determining factors in the formation of microstructure. Therefore, in order to control and manipulate such properties, the study of product microstructure, and in particular, how the microstructure will be influenced by the composition and the processing conditions, is of vital importance.

In this paper, we investigate how the conditions of shear, crystallization, and deformation effect the product microstructure, in particular, the fat crystalline matrix and emulsion structure. We further investigate the relations with product properties.

#### Methods

Fat spreads are processed commercially in the A and C-units of a votator line (2,7). An A-unit consists of a scraped-surface tube cooler in which mixing of the fat and water phase, as well as, partial crystallization of the fat phase occurs, depending on the temperature at the exit of the unit (ex A-temperature). Important parameters are the throughput and the rotational speed, presenting conditions of shear in the early stages of the manufacturing process. After supercooling and partial crystallization in the A-units, further crystallization may occur in the C-units. A C-unit is a cylinder fitted with pins on its inner wall and on the rotor. Application of such a unit induces conditions of strong working.

Shortenings are prepared by processing a fat blend without water phase in a votator line.

The influence of process conditions on the microstructure has been studied in a laboratory votator with about 3 kg of fat, by varying the ex-A temperature and the rotational speed of the A-unit. The effect of a C-unit on microstructure has been investigated by comparison of samples, processed in an A-A and an A-A-C sequence.

Hardness is measured after storage of the products by means of a cone penetrometer (1).

Some samples were rheologically characterized in uni-axial compression. From the margarine discs



Fig. 1. Microstructure of shortenings. a. Complete crystallization in processing line. b. Partial crystallization in rest.

of 45 mm diameter (with ratios of diameter to height between 3 and 10) were cut using a wire. The samples were compressed between thermostated, approaching parallel plates of 45 mm diameter, using a computer controlled Instron testing machine model 1122. This represents a situation of uniform compression in a macroscopic sense. Stress-strain curves of the material were obtained by eliminating the contribution of friction between the sample and the compressing plates, by performing compression tests with samples of different heights. From such a curve (see Results and Discussion, Fig. 2) three characteristic parameters can be deduced: the maximum stress  $\sigma_{\mbox{max}},$  the deformation at maximum stress  $\varepsilon_{max}$  and the ratio ( $\sigma_{rat}$ ) of the stress at large deformation  $(\sigma_{\infty})$  and  $\sigma_{max}$ . The latter ratio is a measure of the work softening, which, in turn, may be related to the breaking of bonds in the product.

The microstructure was analyzed by Cryoscanning electron microscopy (SEM) after de-oiling the products (3) at  $10^{\circ}$ C. For the shortenings and the margarines, a de-oiling time of 28 hours and 20 hours respectively, was applied. Solid phase contents were measured by pulse nuclear magnetic resonance technique.

#### Results and Discussion

#### Shortenings

Influence of cooling regime. A model fat blend, containing 14% of a high melting palm oil fraction (melting point 58°C) in sunflower oil, has been processed in a A-unit. A low ex-A temperature (10°C) resulted in complete crystallization of the solid phase (14%) in the A-unit. At a high ex-A temperature (28°C) only partial crystallization of the solid phase (6%) in the A-unit occurs. The remaining part (8%) of the solid phase crystallizes in rest. The microstructure of the solid fat phase is presented in Fig. 1 which shows that complete crystallization in the A-unit results in a homogenous microstructure of small connected plate-like crystals, whereas partial crystallization in rest leads to a nonhomogeneous structure of large clusters interconnected by crystal bridges.

Both products have also been investigated by parallel plate compression. The stress-strain curves (Fig. 2) are in good agreement with the observed microstructure, considering that on deformation more bonds will be broken in the product with the homogeneous microstructure (Figs. 1a and curve a in Fig. 2) than in the other product (Figs. 1b and curve b in Fig. 2), where only crystal bridges between the clusters will be broken. The product with the homogeneous microstructure has the highest value of  $\sigma_{max}$ (greater hardness) and shows the greatest work softening ( $\sigma_{rat} = 0.08$  whereas the other product has an  $\sigma_{rat}$  of 0.41).

Influence of deformation. In order to obtain better insight into the effects of deformation, the microstructure of the model fat blend, processed via an A-unit and partially crystallized in rest, has been further investigated before and after uniform compression between parallel plates. Before deformation (Fig. 3a) the structure is composed of crystalline clusters interconnected by a fat-crystalline network. After deformation (Fig. 3b) the structure between the clusters is more open. Crystal bridges have apparently been broken and the interconnecting network is, at least partly, being removed. The stressstrain behavior for this product is given in Fig. 2 (curve b).

On the basis of this information the following picture emerges: the region where only elastic deformation occurs is extremely small. Already at very low deformation, breakdown of crystal bridges between clusters occurs and both elastic deformation and fracture take place. At high deformation, after breakdown of the bonds, the deformation is a plastic one, depending on smoothness, shape, and elasticity of the clusters.

Influence of shear. The influence of C-unit (conditions of strong working during fat crystallization) is illustrated in Fig. 4. When the supercooled fat blend is worked in a C-unit, the samples obtained show a more granular structure than samples exclusively processed in A-units (which show a more continuous structure). This observation agrees well with the idea that during working in a C-unit continuous structure formation is prevented, as well as with the

#### Effect of Processing on Microstructure of Fat Spreads

Fig. 2 (at right). Stress strain curves obtained from parallel plate compression after elimination of friction.  $h_0$  and h: height of sample before and after compression respectively. Products shown in Fig. 1 (T = 20°C). a. Complete crystallization in processing line.

- b. Partial crystallization in rest.

Fig. 3 (at center of page). Influence of deformation (parallel plate compression) on microstructure.

- a. Before deformation, presence of crystal bridges;
- After deformation, absence of crystal b. bridges.

Fig. 4 (at bottom of page). Influence of shear (C-unit) on microstructure of shortenings.

- a. Without shear (A-A units only), continuous structure;
- With shear (A-A-C processing), granular b. structure.











finding that A-A-C processed samples are much softer than A-A processed samples (hardness at 10°C of 600 g/cm<sup>2</sup> and 2900 g/cm<sup>2</sup> respectively). It should, be realized that the large grains (up to 40 µm) visible in Fig. 4b would not survive a C-unit and are apparently formed after the processing in the C-unit. Postcrystallization is responsible for this phenomena; this is indicated by the influence of the ex-A temperature on granularity: at high temperatures granularity is more prominent. In this respect there is a resemblance with the structure observed after crystallization in rest (Fig. 2, curve b). Margarines

Microstructures of spreads containing 20% water after A-A and A-A-C processing are given in Fig. 5. A very distinct influence of the C-unit is observed:

- the fat structure is much more granular, as was observed for shortenings;
- shell formation around water droplets is more pronounced.

In addition, larger water droplets are observed (coarsening). Also in this case A-A-C processed samples are much softer than A-A processed samples (hardnesses at 10°C of 600 g/cm<sup>2</sup> and 2300 g/cm<sup>2</sup> respectively). Apparently the influence of the water phase on product hardness is limited.

The better shell formation around the water globules on working may be connected with the enhanced possibility of transport of fat crystals to the oil-water interface. It has been discussed (4-6) that stirring strongly accelerates the rate of adsorption of crystals onto the emulsion droplets. A similar effect is induced by the presence of surfactants (5). When no stirring or working is applied, or in the absence of an emulsifier, free diffusion of crystals will be hindered due to the rapid formation of a solid crystalline network. Consequently the main part of the crystals will stick together to form a network and will not be available for this so called Pickering stabilization of the emulsion droplets. When the emulsion is worked in a C-unit, however, during the crystallization stage crystals will adhere to the emulsion droplets and form a crystalline shell around the water droplets. In addition, smaller crystals, induced by working, can better accommodate and consequently better adhere to the droplet surface.

The coarsening of the emulsion droplets after C-unit processing may also be ascribed to the influence of working. As a result of fat crystallization (in particular in the applied C-unit), the viscosity increases, which may lead to deformation of droplets and subsequent coalescence. Coalescence under the influence of a C-unit is, however, not always observed.

In contrast, a strong shear influence on droplet size can be realized by varying the shear rate in the A-unit. Under conditions of high shear (Fig. 6a) a much finer emulsion is formed than at low shear (Fig. 6b). This difference in behavior should be ascribed to the more liquid-like character of the emulsion in the A-unit, which is still in a supercooled acrystalline state (2). In this case, higher shear just gives a stronger emulsification; coalescence of droplets, due to a high viscosity of the continuous phase as observed in applying a C-unit, does not occur.

#### Concluding remarks

The microstructure of fat spreads can be manipulated and controlled by the applied processing. Microstructure and perceived macroscopic properties appear to be related. This opens up the possibility to intentionally prepare products with desired physical and sensorial properties.

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#### Discussion with Reviewers

D.P. Dylewski: How does fat crystal distribution and shell formation around water globules in margarine change with time?

Authors: We cannot give a general answer to this question. It is highly dependent on blend composition and storage regime. On heating and subsequent cooling of certain fat blends (so called cycling), fractionation effects are observed, in general accompanied by coarsening of fat crystals and changes in water droplet structure.

K. Sato: Do you think that different polymorphic modifications of triacylglycerols such as  $\alpha$ ,  $\beta$ , and  $\beta'$  are present in the structures presented in your figures?

Authors: By X-ray diffraction techniques we found the predominant modification in fat spreads to be  $\beta'$ (see Jurianse and Heertje, this issue). In special cases  $\beta$ -crystals may be found, sometimes giving rise to such adverse properties as sandiness. During processing (rapid cooling!) first the a-modification is formed, which is, in general, rapidly converted to  $\beta'$ modification. Effect of Processing on Microstructure of Fat Spreads



- Fig. 5. Influence of shear (C-unit) on microstructure of margarines.
  - a. Without shear, continuous structure, fine emulsion, no shells around interface; b. With shear, granular structure, a more coarse emulsion, shells around interface.



- Fig. 6. Influence of shear (rotational speed of A-unit) on microstructure of margarines. a. Rotational speed 600 rev./min., fine emulsion;
  b. Rotational speed 100 rev./min., coarse emulsion.

