Although anhydrous milk fat (AMF) has excellent properties, its variable physicochemical properties and its lack of functionality restrict its uses in the food industry. A technology involving dry fractionation of AMF has been developed, and its attributes include selectivity, reliability and general application. Combining two simple and reliable technologies, i.e., multi-step fractionation and blending, it is possible to overcome functionality problems and the seasonal variations of AMF.

KEY WORDS: Blending, crystallization, differential scanning calorimetry, dry fractionation, fatty acid profile, milk fat, spreadable butter, triglyceride composition.

Anhydrous milk fat (AMF) does have advantages, including incomparable organoleptic qualities and reputation as a natural product. In certain high-quality foods and in other applications, butter or milk fat is required for flavor. However, milk fat also has disadvantages. First of all, its chemical composition varies greatly with the season, the region, the breed of dairy cow and the animal feed used. Its physicochemical properties are therefore too variable. Moreover, its lack of functionality for the commercial user (for instance, plasticity and hardness for puff pastry) and for the consumer (for example, poor spreadability of refrigerated product) restricts its potential uses in the food industry. Many of the functionality problems and seasonal variation of milk fat may be completely or largely overcome by fractional crystallization.

The purpose of melt crystallization, or simply “dry fractionation,” is the separation of triglycerides on the basis of their melting points. Schematically, single-step fractionation yields a hard fraction called stearin and a soft or liquid fraction called olein. The melting points or dropping points (D. pt.) (AOCS Method Cc 18-80; Ref. 1) may range from 40 to 46°C for stearins and from 18 to 28°C for oleins, depending on the filtration temperature.

Today, in Europe, over 800 tons per day of milk fat are fractionated with the Tirtiaux dry fractionation process (2-4). This technology involves two main steps, crystallization and separation. On an industrial scale, milk fat is heated and then cooled in stainless-steel crystallizers equipped with a cooling coil and a variable-speed agitator. Monitoring and controlling the crystallization are extremely accurate with the help of computerized process controls.

The separation of crystals is either carried out under vacuum on a Florentine filter or under pressure on a membrane filter. In the Florentine, filtration takes place horizontally and continuously on an endless rotating, stainless-steel perforated belt under slight vacuum. The filter is self-cleaning and the filtration area is enclosed and air-conditioned. A recycling device enables the first filtered olein to be recycled.

Filtration may also be done on a membrane filter, which looks like a plate-and-frame filter, except that each chamber is equipped with a membrane made of flexible material. When the chamber is full of stearin, the membrane is inflated, thereby increasing the pressure up to 4 bar. This enables most of the remaining liquid or olein to be squeezed out of the crystallized fat in the chamber.

This paper presents a general survey on melt crystallization control, crystallization behavior of milk fat, monitoring of dry fractionation by differential scanning calorimetry (DSC), selectivity of dry fractionation in terms of triglyceride groups, efficiency of the process and specific applications.

MELT CRYSTALLIZATION CONTROL

Four factors have to be taken into account to achieve good crystallization from a melt: the technique, oil composition, inter solubility and polymorphism (5).

The technique involves the design of the crystallizer and the parameters of crystallization. It has to control two main steps, nucleation (the birth of new crystals) and growth. The driving force behind nucleation is the supersaturation of the melt, which can be achieved by a progressive lowering of the temperature. The second step, i.e., the growth of existing crystals, requires that the cooling rate as well as the agitation rate must be perfectly controlled to ensure good heat transfer through the mass and excellent solute transfer between crystals and mother liquor.

Crystallization of fat is always complicated and is influenced by many factors, but crystallization of milk fat is even more intricate than that of most other fats because of its complex composition. Milk fat contains more than 40 different fatty acids, with around 70% saturated acids. These saturates are made up of 25% short-chain acids and 45% long-chain acids. Besides that, we find 27% unsaturated and about 3% polyunsaturated fatty acids. Due to its large number of fatty acids, a huge number of triglycerides are present (6-11). They may be distributed into two main groups, one with carbon numbers from C26 to C42 (the lighter triglycerides) and the other from C44 to C54 (the heavier triglycerides). C42 remains the point of intersection as it is relatively constant. Milk fat can be manufactured from fresh cream, either directly or indirectly via butter, or from stored butter.

Crystallization is considerably influenced by inter solubility (12,13); the phenomenon of co-crystallization and the formation of mixed crystals, which contain more than one triglyceride species, is common in milk fat. It must be remembered that crystallization occurs in a finite time interval and that structures are consequently set up whose origins are of kinetic rather than thermodynamic nature.

Superimposed on the complexities of mixed crystals is the phenomenon of polymorphism. Triglycerides can crystallize into different crystalline forms, called α, β and β ′, with increasing stability and melting points.


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All the factors mentioned here are so interdependent that one can consider milk fat crystallization a "technique as well as an art."

CRYSTALLIZATION BEHAVIOR OF MILK FAT

Going through the crystallization behavior of milk fat, we will analyze the selectivity of dry fractionation with respect to physical characteristics and triglyceride compositions of the fractions.

The DSC melting profile of a typical whole milk fat clearly shows three major peaks due to three major groups of glycerides melting independently (Fig. 1): a low-melting fraction (LMF) from $-25$ to $+10\,^\circ C$; a middle-melting fraction (MMF) from $10$ to $19\,^\circ C$ with a pronounced peak at $17\,^\circ C$; and a broad high-melting fraction (HMF) from $19$ to $34\,^\circ C$. The relative size and position of the peaks vary with the thermal history of the fat and its triglyceride composition (14,15).

DSC can be used to monitor the dry fractionation of milk fat. For example (Fig. 2), the olein fraction from a single-step $18\,^\circ C$ fractionation (designated as olein 18) only registers two peaks in its DSC profile: an LMF from $-26$ to $12\,^\circ C$ and an MMF from $12$ to $19\,^\circ C$. The HMF is thus completely removed at $18\,^\circ C$. The maxima have shifted to $10$ and $16\,^\circ C$. However, for oleins obtained at a higher temperature than $18\,^\circ C$, the thermogram shows a shoulder on the right side, and melting of the MMF and LMF has been altered substantially.

Looking now at the stearin, the HMF is of course the major peak with a maximum at $41\,^\circ C$ and a minimum at $20\,^\circ C$. Beside the main peak, two minor peaks appear at $7$ and $16\,^\circ C$, corresponding respectively to the LMF and MMF. The presence of these peaks is due to olein entrained in the stearin. The major part of olein occluded in the stearin may be removed by filtering the slurry on a membrane filter (Fig. 3), in that case, the DSC melting curve mainly consists of one peak, the top of which appears at about $46\,^\circ C$.

**FIG. 1.** Differential scanning calorimetry melting profile of milk fat. Abbreviations: LMF, low-melting fraction; MMF, middle-melting fraction; HMF, high-melting fraction. Conditions: heat to $60\,^\circ C$, hold for 5 min, cool at $5\,^\circ C/min$ to $-50\,^\circ C$, hold at $-50\,^\circ C$ for 5 min, heat to $60\,^\circ C$ at $5\,^\circ C/min$.

**FIG. 2.** Differential scanning calorimetry melting curves of milk fat fractions (first step) [milk fat (D. pt. $32\,^\circ C$)] derived from milk fat shown in Figure 1. Florentine filter was used for separation. D. pt. is dropping point.

**FIG. 3.** Differential scanning calorimetry melting curve of milk fat stearin (single step) obtained from membrane filtration. Compare to stearin in Figure 2. See Figure 2 for abbreviation.