Milk Fat Fractionation Today: A Review[1]

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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, intersolubility 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 intersolubility (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°C; a middle-melting fraction (MMF) from 10 to 19°C with a pronounced peak at 17°C; and a broad high-melting fraction (HMF) from 19 to 34°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°C fractionation (designated as olein 18) only registers two peaks in its DSC profile: an LMF from −26 to 12°C and an MMF from 12 to 19°C. The HMF is thus completely removed at 18°C. The maxima have shifted to 10 and 16°C. However, for oleins obtained at a higher temperature than 18°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°C and a minimum at 20°C. Beside the main peak, two minor peaks appear at 7 and 16°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°C.

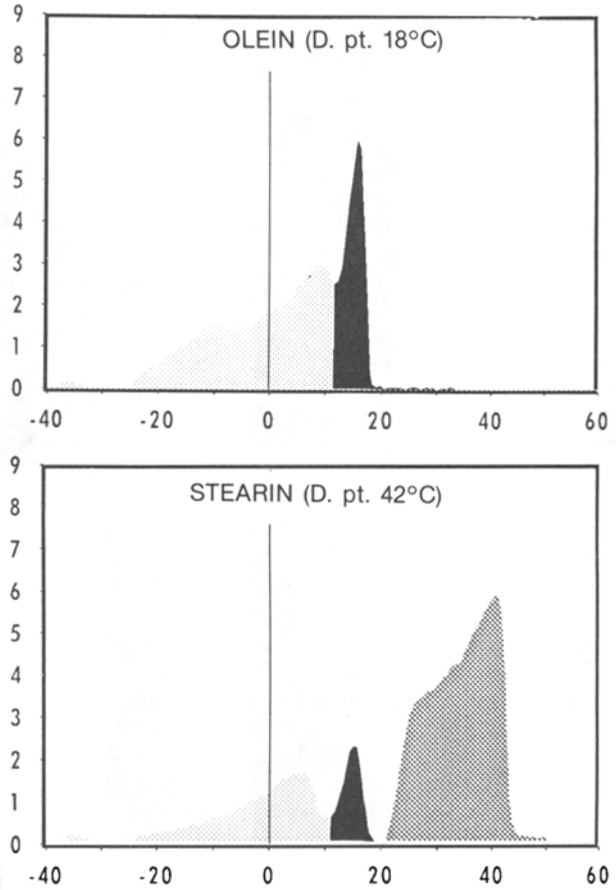

FIG. 2. Differential scanning calorimetry melting curves of milk fat fractions (first step) [milk fat (D. pt. 32°C)] derived from milk fat shown in Figure 1. Florentine filter was used for separation. D. pt. is dropping point.

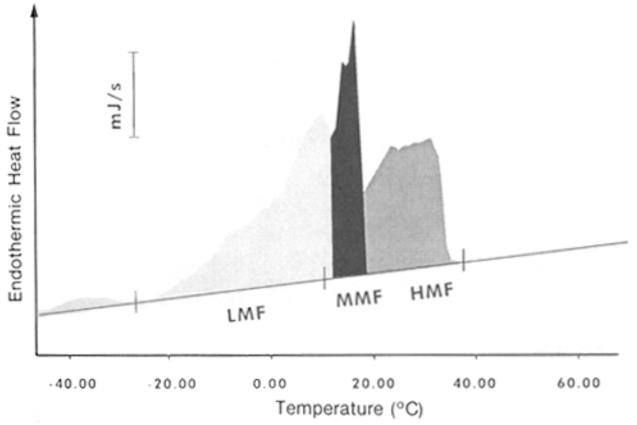

FIG. 1. Differential scanning calorimetry melting profile of milk fat. Abbreviations: LMF, low-melting fraction; MMF, middle-melting fraction; HMF, high-melting fraction. Conditions: heat to 60°C, hold for 5 min, cool at 5°C/min to −50°C, hold at −50°C for 5 min, heat to 60°C at 5°C/min.

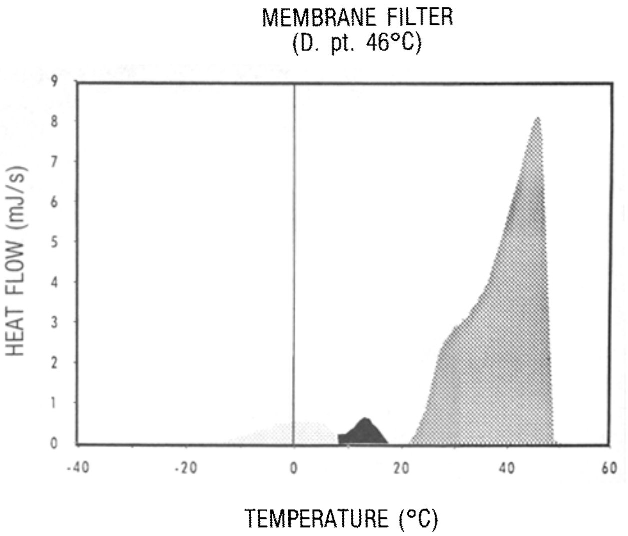

FIG. 3. Differential scanning calorimetry melting curve of milk fat stearin (single step) obtained from membrane filtration. Compare to stearin in Figure 2. See Figure 2 for abbreviation.

Further fractionation of olein 18 below 10°C yields a second olein, which only registers one peak in its DSC profile (Fig. 4). Removal of MMF results in a melting curve that is similar to the lowest melting peak of original milk fat. However, oleins whose melting points are higher than 9°C do not differ in thermal behavior from original olein, except that there is sharper melting (narrower peak) with a shift of the maxima towards lower temperatures. On the other hand, the soft stearin is particularly enriched in MMF.

When comparing the percentage of LMF, MMF and HMF in each thermogram, we observe excellent selectivity in terms of melting properties. Figure 5 shows that: (i) a pure LMF can be successfully obtained out of milk fat in two steps; (ii) there is a good relationship between the percentage of super-olein obtained and the percentage in LMF in original milk fat; (iii) the purity in LMF can be guaranteed only for olein of 10°C D. pt. maximum; otherwise it will be contaminated by MMF as in the first olein fraction; (iv) compared to olein 18, soft stearin presents a total inversion of LMF and MMF. Its contamination with LMF depends in particular on the type of separation; (v) there is a strong enrichment of HMF in the stearins, especially for the hard stearin from a membrane filtration. In practice, a 10% yield gap is registered between the two stearins. This difference is mainly due to their relative contamination in LMF and MMF from occluded olein; (vi) the HMF are probably made up of high-melting glycerides associated with other glycerides in mixed crystals with the same polymorphism and, hence, sharing the same peak. Their proportion depends on crystallization rate (parameters) and fat composition. Therefore, it is of interest to examine the chemical composition of these fractions.

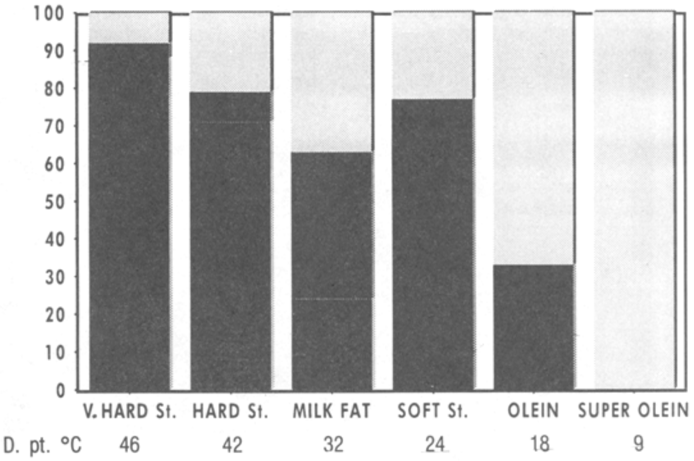

FIG. 5. Peak areas of differential scanning calorimetry thermograms of milk fat and selected fractions (St. = stearin; V = very). See Figures 1 and 2 for abbreviation. HMF = medium-dark shaded bars; MMF = dark-shaded bars; LMF = light shaded bars.

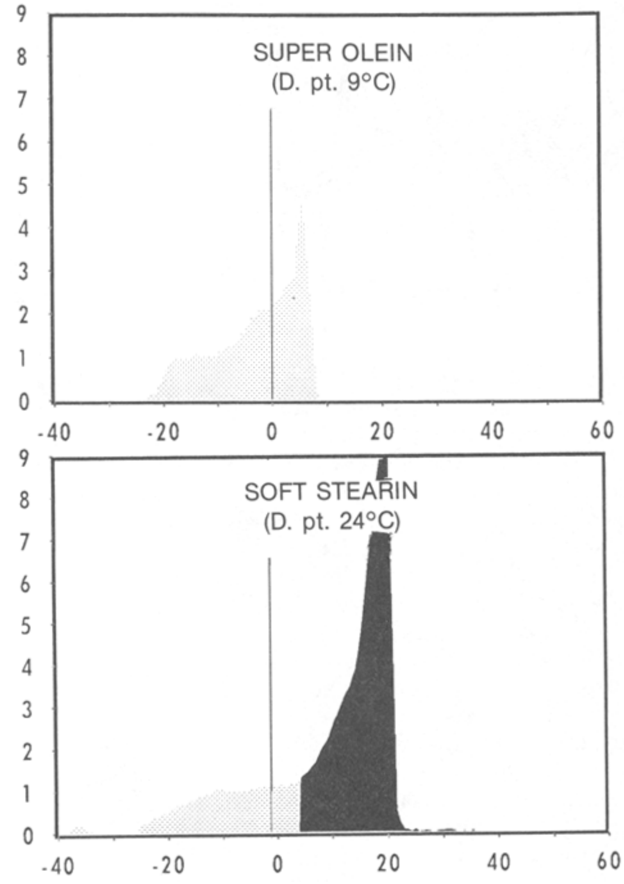

FIG. 4. Differential scanning calorimetry melting curves of olein fractions (second step) [olein (D. pt. 18°C)]. See Figure 2 for abbreviation.

Fatty acid composition. When comparing the fatty acid composition of milk fat and that of very hard stearin, the first impression is that we are comparing the fatty acid compositions of soft and hard butter where palmitic, stearic and oleic acids vary the most (Fig. 6). Considering the great difference in melting points between these two samples (32 and 46°C), increases of 8 and 7% in palmitic and stearic acids, as well as a decrease of 8% in oleic acid, are not as large as we may expect.

If we compare the fatty acid composition of milk fat with that of super olein (Fig. 7), the variation in palmitic, stearic and oleic acids is quite similar but totally inverse.

The distribution of saturated and unsaturated fatty acids in these fractions reveals that there is a 10% decrease of unsaturated acids in the stearins and, inversely, a 10% increase of unsaturated acids in super olein. Reading the literature on dry fractionation, one discovers the following comment: "fractions have very different physical properties but they are still maintaining an overall similarity in gross fatty acid composition" (16). It is clear that the fatty acid composition is not the best criterion to judge the selectivity of dry fractionation of milk fat. This may be explained by the fact that the physical properties of milk fat fractions depend on both fatty acid composition and their distribution on the glycerol.

Triglyceride composition. Milk fat triglyceride analysis has been accomplished by high-performance liquid chromatography (HPLC) on a silver-loaded column, according to the method published by Christie (17,18), with a light scattering detector. The peaks were identified and quantitated after collection by adding methyl monadecanoate as an internal standard and transmethylating before gas-liquid chromatography analysis. This method enables the

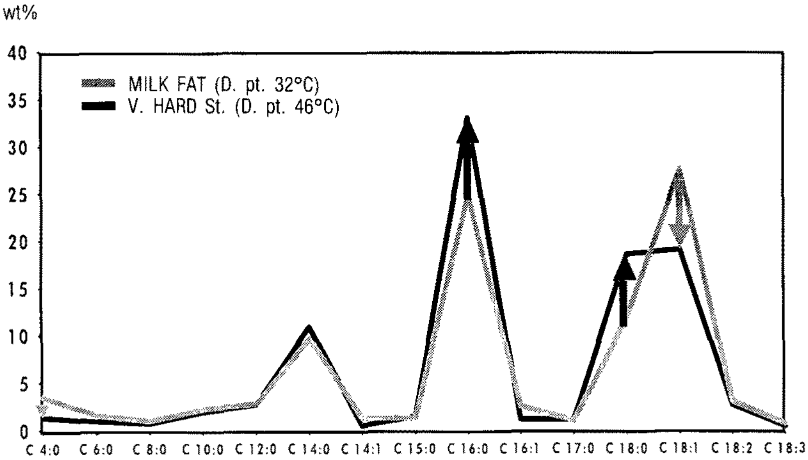

FIG. 6. Fatty acid compositions of milk fat and very hard stearin. For abbreviations, see Figures 2 and 5.

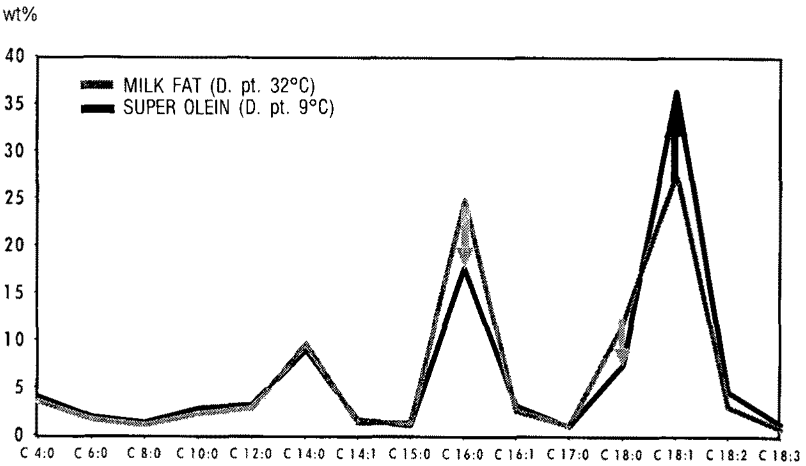

FIG. 7. Fatty acid compositions of milk fat and super olein. D. pt., dropping point.

separation of milk fat into triglyceride groups with zero, one, two and three unsaturations in the fatty acyl chains. Figure 8 displays the three main groups: SSS, SSU (with SSE and SSM included for simplifaction), and UUU-SUU, where UUU is associated to SUU because of its low content [S, saturated; U, unsaturated; E, elaidic (*trans*-monoenoic); M, *cis*-monoenoic].

By complementary gas chromatography analysis, each group has been quantitated in two subgroups (Fig. 9), the lighter triglycerides from C24 to C42 and the heavier ones from C44 to C56. It appears then that milk fat contains around 50% monounsaturated triglycerides (SSU), with lighter and heavier triglycerides in about equal concentration. The second group, SSS, mainly contains lighter triglycerides. In the third group, SUU-UUU, the heavier triglycerides are slightly dominant. This triglyceride composition by groups of unsaturation goes along quite well with the analytical results obtained by Kankare and Antila (19) and Taylor and Hawke (20) who have used other techniques to quantitate similar milk fat samples.

A direct relationship generally exists between triglyceride composition and physical properties of a fat. In milk fat, the situation is more complex because of its large number of different triglycerides. However, when comparing the triglyceride composition of milk fat and of selected fractions (Fig. 10), one can perceive a clear cut: heavier saturated triglycerides (C44–C56) are almost completely removed from the olein, whereas they are highly concentrated in the stearins.

On the other hand, lighter saturated triglycerides (C24–C44) are strongly reduced in the stearins but remain unchanged in the olein. The monounsaturated group shows the same trend: the lighter triglycerides are strongly reduced in the stearins and significantly increased in the olein and the inverse is true for heavier triglycerides. Parallel to that, there is a strong enrichment of heavier diunsaturated triglycerides in the super olein. Such a trend is further enhanced in olein of D. pt. 6°C. Figure 10 shows that the presence of lighter triglycerides in stearins is due to contamination by occluded olein. Most of it can be eliminated by membrane filters but there is some left, as shown initially by DSC. On the contrary, the presence of heavier monounsaturated (SUS) and diunsaturated (SUU) triglycerides may be explained by the fact

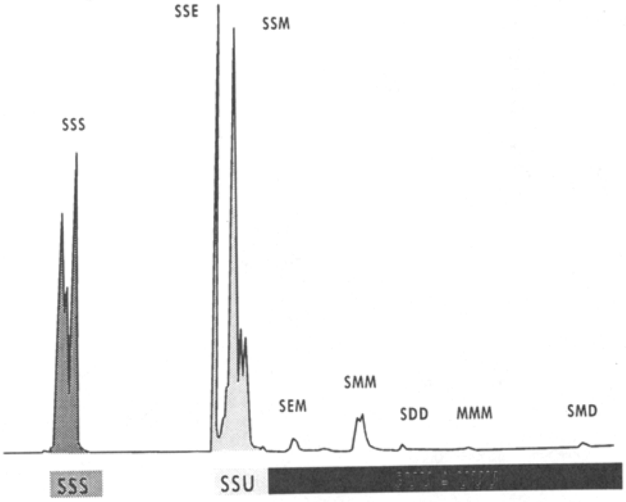

FIG. 8. Separation of molecular species of milk fat triglycerides by silver ion high-performance liquid chromatography. Abbreviations: S, saturated; E, elaidic (*trans*-monoenoic); M, *cis*-monoenoic; D, dienoic; U, unsaturated. Conditions: column (250 × 4.6 mm i.d.) Chromspher Lipids from Chrompack (Middelburg, The Netherlands). Mobile phase, A, dichloromethane/dichloroethane 50:50 vol/vol; B, acetone. Gradient O → 3 min: 100% A; 3 → 43 min 100% A → 50% A + 50% B. Detector, ACS Model 750/14 (ACS, Macclesfield Cheshire, United Kingdom). Flowrate 1 mL/min. Temp. 20°C.

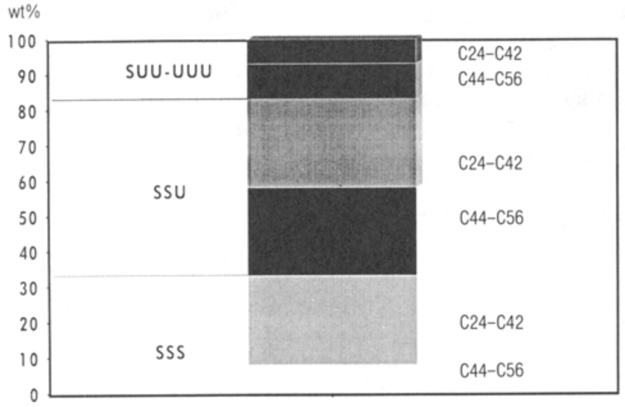

FIG. 9. Triglyceride composition of milk fat (wt%). Abbreviations: C24-C42, lighter triglycerides; C44-C56, heavier triglycerides. See Figure 8 for more abbreviations.

that they are considerably associated with the heavier trisaturated triglycerides in mixed crystals (13).

EFFICIENCY

Considering the physical and chemical characteristics of milk fat fractions, we can evaluate the efficiency of dry fractionation in terms of step number and mass balance. To get a super olein, the fractional crystallization will require either two or three steps. As originally stated, a super-olein with a D. pt. of 10°C maximum can be obtained from a soft butter directly in two steps (Fig. 11), the first filtration taking place at 18°C. The olein of 18°C D. pt. will then be recrystallized and filtered to give a middle stearin and a second olein. The overall olein yield is around 30%.

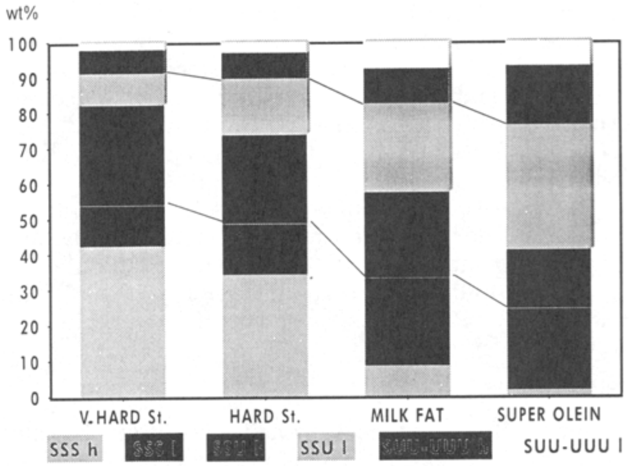

FIG. 10. Triglyceride composition of milk fat and selected fractions (wt%). Abbreviations: see Figures 9 and 5.

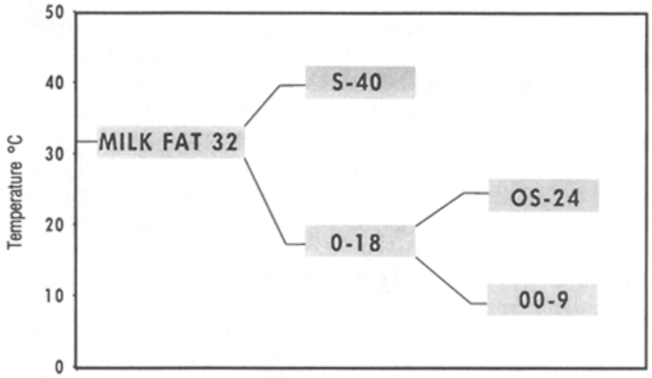

FIG. 11. Scheme of double fractionation of milk fat. Abbreviations: S, stearin; O, olein; OS, 2nd stearin; OO 2nd olein. Drop points of fractions are indicated behind letter designations.

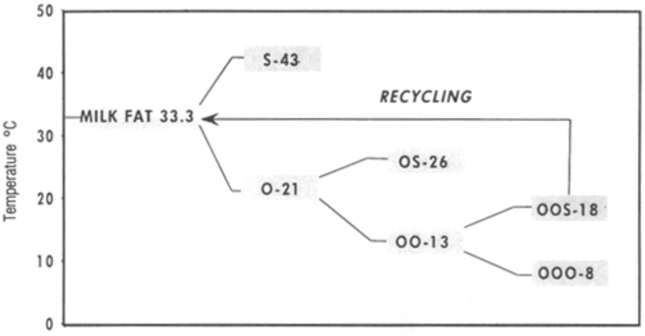

FIG. 12. Scheme of triple fractionation of milk fat. Abbreviations: see Figure 11.

Starting with a hard milk fat (D. pt. 33°C), the scheme of fractionation (Fig. 12) is different because an olein of 18°C D. pt. is quite difficult to get in one step. Therefore, the last part of the high-melting triglycerides will be eliminated in the second step, while the MMF of milk fat will be removed in the third step. The minimum overall olein yield is 20%, but it can be increased either by the use of the membrane filter as shown earlier and/or by recycling the soft stearin of 18°C D. pt. in the feed. This fraction represents 20–25% of overall yield and has little application *per se.*

It is interesting to compare our scheme with those published in the literature where five steps are generally required, as mentioned by Guyot (21) or Makhlouf *et al.* (22). Such a multiple-step fractionation process is uneconomical from an industrial point of view.

APPLICATIONS

Fractionation has the most dramatic effect on the solid fat content (SFC) profile. Therefore, the general application (23,24) of milk fat fractions will be correlated to their SFC curves. Milk fat fractions can be either used as such, or they can be blended in various proportions for use as ingredients in various food fat formulations.

Stearins. Stearin fractions with a D. pt. range from 40 to 46°C are used in three main applications: shortenings, confectionery and as hardstock. A shortening will have a flat SFC profile over 10 to 25°C to provide a wide plastic range (Fig. 13). Shortenings are principally used for the manufacture of "puff pastry" (D. pt. 42–46°C), Danish pastries and croissants (D. pt. 38°C). Excellent results can be obtained that are comparable with shortenings and margarines tailor-made for the same applications. Milk fat stearin fractions also show enhanced flavor. For most applications, milk fat fractions have to be plasticized for optimal performance (25).

In confectionery, milk fat is an important ingredient. The hard fraction is more compatible with cocoa butter than normal milk fat. Therefore, when using stearins, more milk fat can be used than otherwise (26).

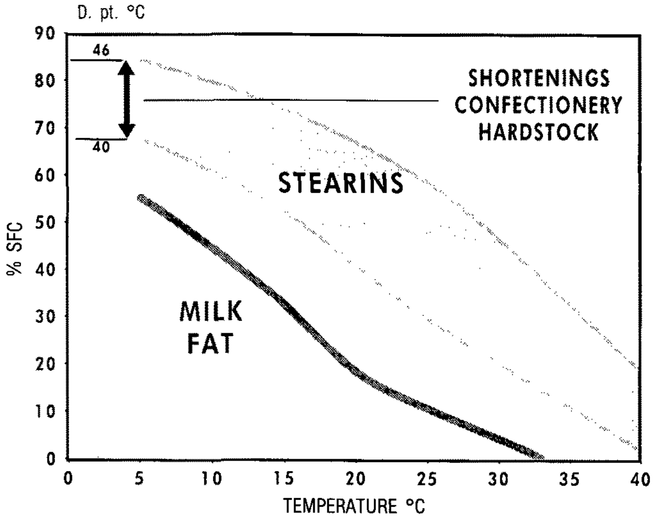

FIG. 13. Solid fat content (SFC) of milk fat and stearins from single stage. Measured by nuclear magnetic resonance on a Bruker PC20 (Karlsruhe, Germany). Conditions: heat to 60°C for 30 min, hold at 0°C for 90 min and then 30 min at the measurement temperature. See Figure 2 for abbreviation.

Additionally, HMF have clearly been shown to inhibit bloom formation more effectively than natural milk fat (27). Stearins can be used as hardstock for ghee production and for reconstitution of hard butters in hot countries or as a component for spreadable butter. Stearin can also act as a hardstock in place of hydrogenated oil when blended with liquid oil. Figure 14 shows the SFC curve of hard stearin blended with soybean oil in a ratio of 20:80. A similar approach has been patented by Unilever (28) in which sunflower and an HMF rather than hydrogenated milk fat are used.

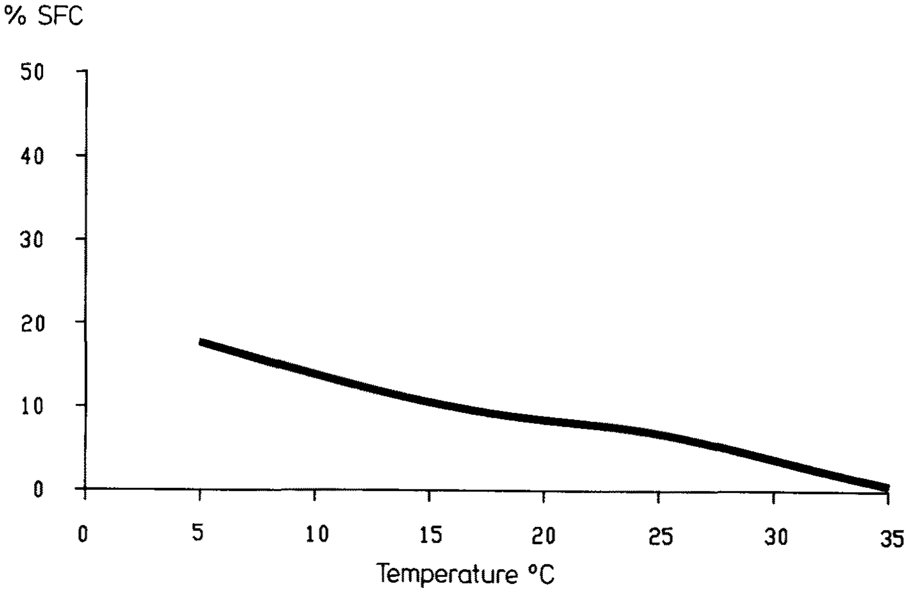

FIG. 14. Solid fat content (SFC) of a blend: very hard stearin milk fat/soybean oil 20:80.

Oleins. Oleins with a D. pt. range from 28 to 6°C (Fig. 15) can be incorporated into milk fat to give a certain mouthfeel or meltdown, for instance, for pound cakes, cookies, etc.

Oleins from single-stage fractionations are mainly used for softening butter, for creaming applications and for spreads. Low-fat (40%) spreads are increasing their market share in many countries. A low-fat dairy spread has poor spreadability and texture if made from plain milk fat. Replacing milk fat by olein (26–28°C D. pt.) gives the lightened butter good spreadability.

Super oleins with a maximum drop point of 9°C are used in any product where fluidity is required at low temperature. One of the most important applications is their use as an ingredient to make spreadable butter. Figure 16 shows that two ways are possible: blending of cream with super olein followed by churning or reconstituting butter with milk fat fractions.

The principle of the first route is to blend the cream and 15 to 25% super olein, based on the total fat content. The new emulsion is pasteurized and then subjected to a special temperature treatment—such as cold-hot-cold (Alnarp-type)—during ripening of the cream. Churning is carried out at 12°C. Figure 17 shows how the hardness is influenced at 6 and 13°C by adding 20% olein to a winter cream (29). All three tests were made at 6-19-9°C

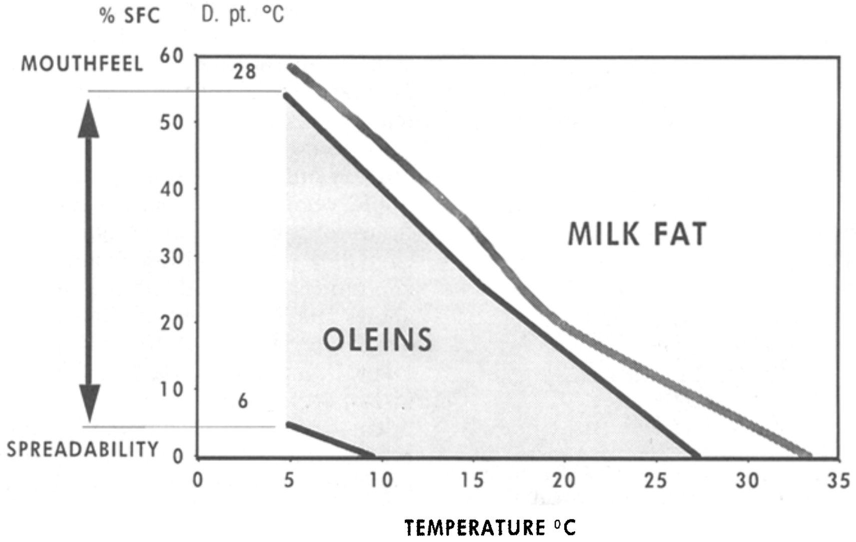

FIG. 15. Solid fat content (SFC) of milk fat and oleins from multi-step fractionation. See Figure 2 for abbreviation.

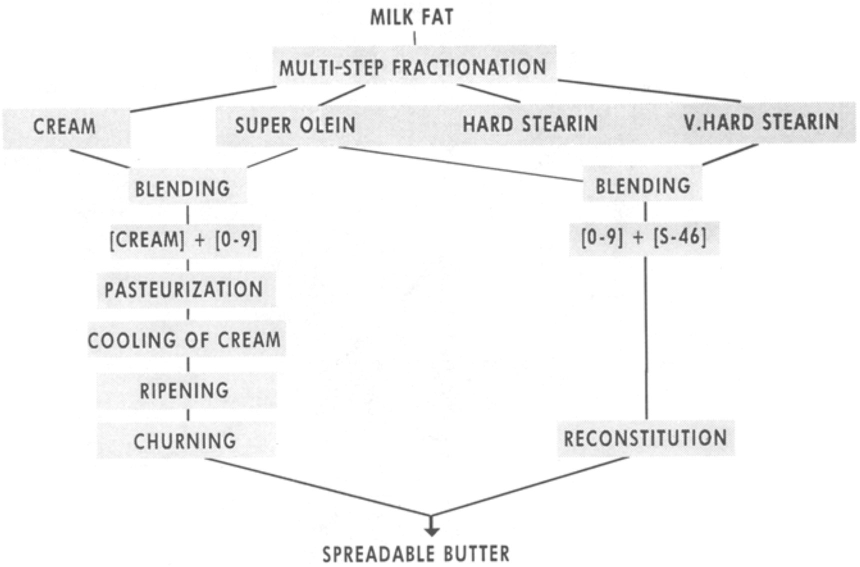

FIG. 16. Production of spreadable butter from milk fat fractions by blending with cream and churning or by reconstitution. See Figure 5 for abbreviation.

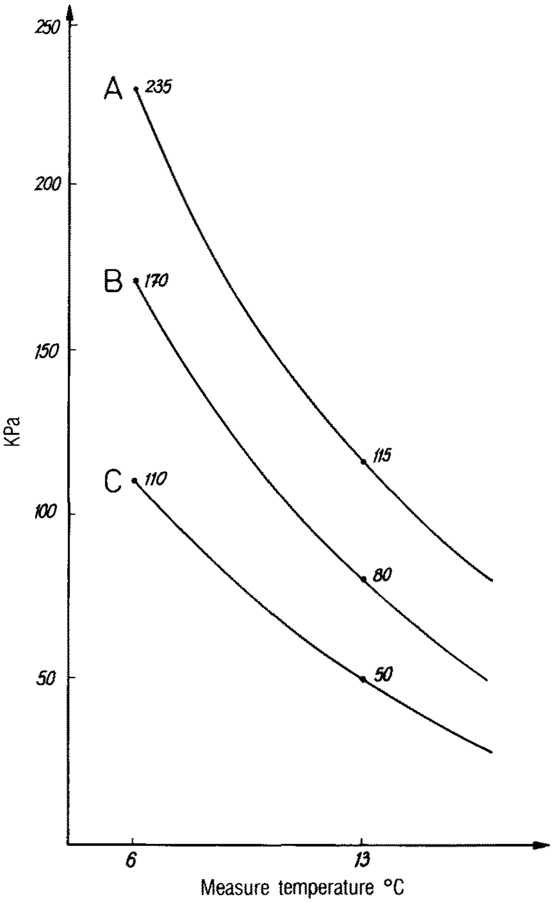

FIG. 17. Influence of fat composition at 6 and 13°C on the hardness of butter. Measured by penetrometer in Kilo Pascal. Curves: A, winter cream; B, 20% olein (16°C dropping point); C, 20% olein (9°C dropping point).

according to the "cold-hot-cold" temperature sequence. Considering a value between 50 and 100 KPa for an excellent spreadable butter, it is clear that good spreadability may be obtained at 6 and 13°C only by mixing olein of 9°C D. pt. into winter cream. Compared to that, the winter cream itself and the other mixture show a totally different pattern. Their hardness is much too high at 6°C. Thus, the strategy adopted consists in combining the effect of temperature treatment of the cream and the utilization of an olein as lubricant; milk fat globules are indeed lubricated by liquid oil, just as a ball bearing.

The second possibility, recognized in the early 1970s, was to recombine the higher and lower melting fractions. That approach has, however, suffered two problems, technology and legislation. Since then, the technology has been improved quite a lot, thanks to the multi-step fractionation development that can lead to suitable products on an industrial scale in two or three steps. The legislation on butter production also changed a few years ago in many countries: fractionation, reconstitution and blending are now permitted. As a result, both kinds of products are presently on the market. The strategy for the second route is similar to that of spreadable margarine made from liquid vegetable oil and hydrogenated oil. The desirable figure in terms of consistency is a solids content lower than 35% at refrigerated temperature (6°C) and yet solid enough (5%) to hold the product up to 25°C. Figure 18 shows the SFC curve of such a blend composed of 20% very hard stearin of 46°C D. pt. and 80% olein of 9°C D. pt. Compared to that, milk fat has a much higher SFC at low temperature.

There are great advantages in combining two simple and reliable technologies, multi-step fractionation and blending (Fig. 19). They generate a wide range of products with standardized specifications and a multitude of possibilities. They correct most deficiencies in functionality, such

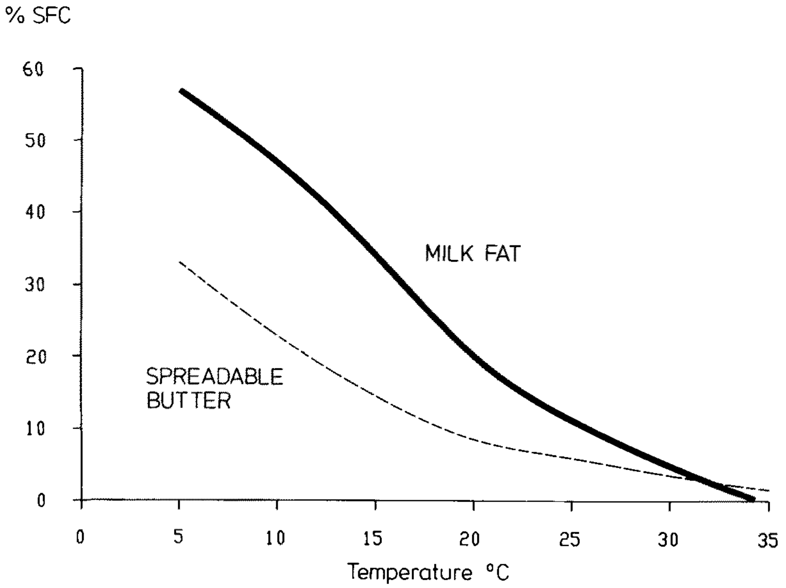

FIG. 18. Solid fat content (SFC) of milk fat and a spreadable butter prepared by reconstitution.

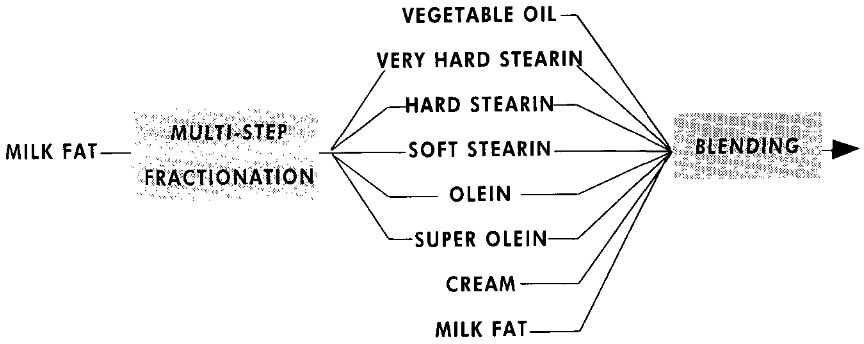

FIG. 19. General process line for milk fat fractionation today.

as lack of consistency and spreadability. Moreover, as the consumption of spreads increases, mixing milk fat fractions into vegetable oils will become the new way when milk fat flavor is desired.

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