Monitoring Milk Fat Fractionation: Effect of Agitation, Temperature, and Residence Time on Physical Properties

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ABSTRACT: With the use of two central composite designs, the effects of agitation rate, fractionation temperature, and residence time on the thermal properties of the stearin and olein milk fat fractions were investigated. The main function of agitation during fat fractionation was suspending the crystal aggregates and enhancing the heat transfer. For the experimental conditions described here, crystal aggregation did not seem to be affected by agitation. The effect of fractionation temperature on the physical properties of the olein fraction was very significant. Triangle diagrams were shown to be a useful tool for monitoring and designing fractionation processes. They illustrate that oleins with similar melting properties can be produced over a range of yields of stearin, which is important from an industrial point of view. Crystallizer residence time, which influences production costs, clearly affects both stearin yield and olein melting properties. For any fractionation temperature, stearin fractions with virtually identical melting properties and yields can be obtained over a range of olein melting properties. Manipulation of both the fractionation temperature and residence time allows the fractionation process to be adapted to meet changing market demands for fractions with different melting properties.

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KEY WORDS: Agitation, milk fat fractionation, residence time, temperature, triangle diagram.

The production of anhydrous milk fat (AMF) was a conservation method for the large stocks of butter produced during the 1980s in the European Community. By removing the water phase from butter, milk fat could be stored for several years without significant loss of quality. Nowadays, AMF is used in the confectionery, bakery, and ice cream industries for its sensorial properties and its marketing value as a natural ingredient. New techniques such as fractionation, texturization, and recombination have led to new applications for AMF, especially in puff pastry and cold spreadable fat blends. These processes are used to improve product quality.

Fractionation is a separation process by which the fat is divided into different fractions, each having its own physical and chemical properties. Two types of fractionation exist: dry (or melt) fractionation and solvent fractionation (1). The latter is never used on an industrial scale for milk fat owing to flavor loss but is extensively used in vegetable oil processing, e.g., for the production of cocoa butter substitutes. In dry fractionation, crystals are formed by controlled cooling and agitation. The crystals in suspension are then separated on a rotary drum belt filter, a filter press, a centrifuge, or by means of an emulsifier followed by a centrifugation step. Fractionation of AMF was extensively reviewed by Kaylegian and Lindsay (2) and more briefly by Deffense (3).

The effects of process parameters such as agitation rate, cooling rate, and fractionation temperature have been investigated several times. Deffense (4) discussed four factors that influence crystallization of milk fat during fractionation: oil composition, polymorphism, rate of cooling, and intersolubility. In addition, the technique used to crystallize milk fat (type of crystallizer, impeller, and operating conditions) can significantly affect the process (5). Several process parameters will be discussed in more detail.

Cooling rate. For the Tirtiaux process (low agitation speed, large volume/cooling surface ratio, and minimal supercooling), decreased crystal size and a more uniform crystal size distribution have been observed with low cooling rates. At these cooling rates, little agglomeration took place, whereas at medium and high cooling rates, more irregular crystal agglomerates were formed (4). In contrast, Herrera and Hartel (6) obtained larger, denser crystals with slow cooling rates and a more uniform crystal size distribution with higher cooling rates. This illustrates that the effect of process parameters is influenced by the working range of the experiments, the type of crystallizer, the type of impeller, and other factors.

Agitation. The effect of agitation is more consistent. Samples crystallized at the highest shear rates produced the smallest crystals and the narrowest particle size distribution (6,7). At low agitation, agglomeration took place, whereas at high shear rates, smaller crystals were formed because of an enhanced nucleation rate (6,8) and breakdown of crystals by the shear forces of the impeller (5,9). Grall and Hartel (8) found the same effect at 30 and 20°C but the opposite at 15°C. This phenomenon was explained by the crystal habit: The uniform spheres formed at 15°C did not seem to be sensitive to shear forces. Patience *et al.* (5) investigated the effects of stirring and type of impeller on filtration properties and crystal size distribution. Breitschuh and Windhab (7) showed that higher agitation promotes cocrystallization, probably due to an enhanced heat transfer. Herrera and Hartel (6) observed that agitation had a minor influence on the induction time of the

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crystallization but that heat transfer and diffusion were increased.

Temperature. Crystallization temperature affects the formation of the various polymorphs, the size of the crystals, the composition of the solid fat, and the physical properties of the fractions (10,11). At higher temperatures fewer initial crystals are formed and their growth is favored, resulting in larger crystals (6). Breitschuh and Windhab (12) showed that a higher degree of supercooling, even for a short period, reduced the difference in composition between stearin and olein as a result of cocrystallization, and this was confirmed by Herrera and Hartel (6).

The goal of the present study was to investigate the effect on milk fat crystallization of several process parameters: agitation speed, end fractionation temperature, and residence time. A new method for process design using triangle diagrams is proposed. This approach helps in understanding the effect of temperature and residence time in a more dynamic way.

MATERIALS AND METHODS

AMF. For each set of experiments, 25 kg of AMF originating from one and the same batch was used to eliminate raw material variability. The AMF was supplied by Aveve Dairy Products (Klerken, Belgium).

Crystallization procedure. The pilot plant crystallizer was constructed by Aveve Dairy Products. The fat was melted in a separate stainless steel jacketed vessel at 60°C for at least 2 h. The melted fat was then transferred to the crystallizer, a stainless steel jacketed vessel that was already heated to 60°C before transfer. During the crystallization procedure, which is a controlled agitation/temperature program, the oil and water temperature were measured on-line and further processed *via* Access (Microsoft). These measurements result in a typical time/temperature profile as displayed in Figure 1. The first 2 h of each experiment were identical. After that the fractionation temperature, the agitation rate, and the residence time (total time of the fractionation) were altered according to a central composite design.

Filtration procedure. At specific time intervals (1–2 h) and again at the final stage, about 400 g crystal suspension was vacuum-filtered on a 0.1 m diameter filter. The yields of stearin, acetone-washed stearin, and olein were determined as the weight fraction of the crystal suspension. These fractions were analyzed by pulsed NMR (pNMR) and DSC.

pNMR. The analysis for solid fat content (SFC) was performed according to AOCS method Cd 16-81 using a Bruker PC20 series NMR analyzer (Bruker, Karlsruhe, Germany) starting from 5°C and increasing the temperature in steps of 5°C.

DSC. Crystallization and melting curves were determined on a TA2010 (TA Instruments, New Castle, DE). The applied time/temperature program was the one described by Deffense (3). In brief, samples were heated to 70°C, held isothermally at 70°C for 5 min, cooled to −40°C at 5°C/min, and heated to 60°C at 5°C/min. Samples were stored for at least 1 h at 60°C to eliminate crystal memory and to allow a homogeneous sampling. Samples were than transferred to aluminum-coated sealed pans with a micropipette. Sample sizes varied between 10 and 15 mg.

Experimental design. The experimental setup was a three-level central composite design. Two sets of 13 experimental combinations were chosen to obtain a relevant statistical survey of the effects of process parameters. The center point was repeated five times. In the first set, agitation and temperature were evaluated, whereas in the second set, the effects of temperature and residence time were investigated. The software Design-

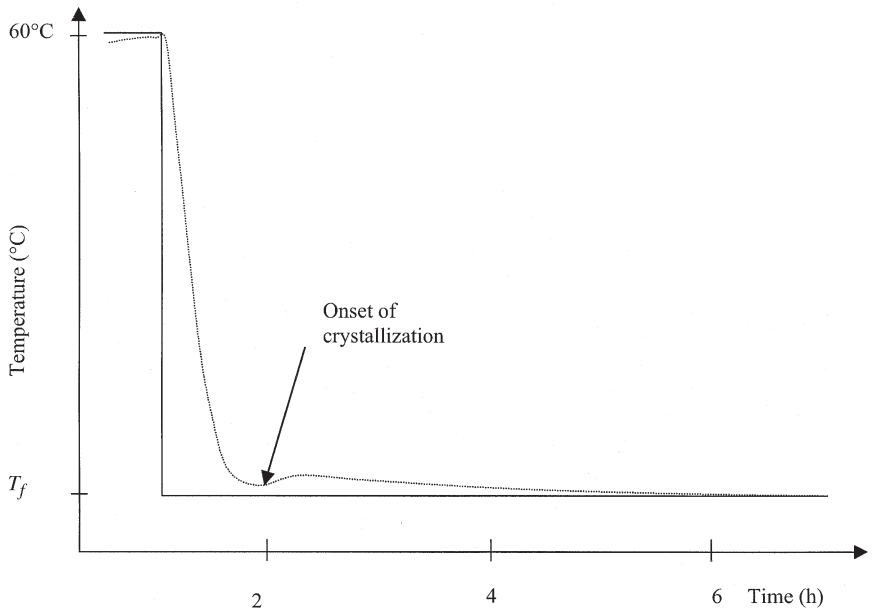

FIG. 1. Typical temperature profile for fractionation. T_f = fractionation temperature; ··· = oil temperature , — = water temperature.

Expert® 5.0 (Stat-Ease, Inc., Minneapolis, MN) was used to fit polynomials to the responses obtained from DSC and pNMR analysis. The final result of these sets of experiments is a response surface within certain ranges of the process parameters. As the uncertainty for an extrapolation is higher than for an interpolation, data for combinations of parameters around the area of interest were gathered. The response surface is described by an equation of the following form:

$$Y = aA + bB + cA^2 + dB^2 + eAB + f \quad [1]$$

The parameter Y is obtained from DSC or NMR analysis, with A and B representing two of the coded process parameters (agitation, temperature, and residence time). The value −1 in coded form was equal to the low level of the process parameter. The value +1 in coded form corresponded to the high level of the process parameter. For example, in the case of residence time, the low level was 6 h, whereas the high level was 12 h. The coefficient f corresponded to the intercept (discussed in the Results and Discussion section). The other coefficients are also included in the tables of results (e.g., c can be found in the table under B^2). To check whether the variation of a parameter of DSC or NMR could be explained as a function of process parameters, an F-test was used. In this case, a P-value of 0.1 was the limit used to retain a parameter for further investigation. With t-tests, the significance of linear, quadratic, and interaction effects of process parameters was tested.

RESULTS AND DISCUSSION

Monitoring milk fat fractionation. In the past, m.p. and FA composition were used for monitoring and optimizing fractionation processes. Nowadays, SFC obtained by pNMR is used for determining fractionation quality. In applications where texture is relevant, it is likely that the proportion of

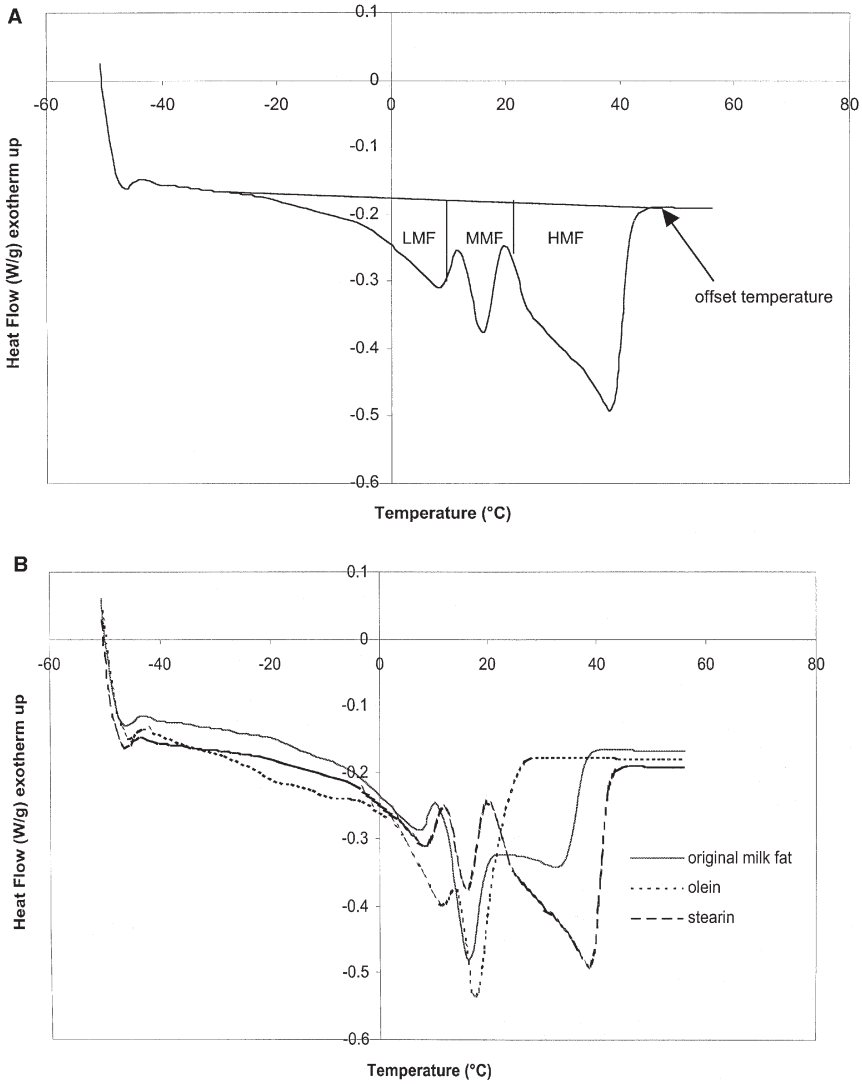

FIG. 2. DSC melting profile of (A) the original milk fat showing different fractional components and (B) original milk fat, and the stearin and olein after fractionation. Scanning rate: 5°C/min. LMF, low-melting fraction (≤10°C); MMF, middle-melting fraction ($10 < T < 21$°C); HMF, high-melting fraction (>21°C).

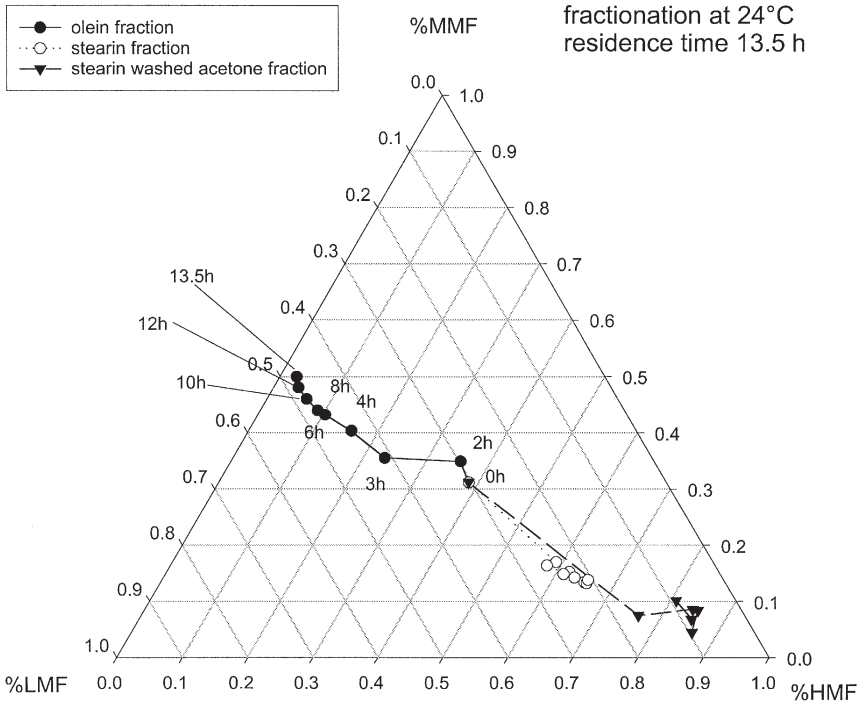

FIG. 3. Triangle diagram based on composition of different milk fat fractions showing changes in composition of solid (stearin) and liquid (olein) fractions during fractionation. For abbreviations see Figure 2.

solid fat at the application temperature is the critical parameter rather than the final m.p. Deffense (3) proposed a method for evaluation of milk fat fractionation based on the DSC melting profile of the fractions obtained. The melting profile from the DSC can be considered as having three parts. Those TG that melt at 10°C and below make up the low-melting fraction (LMF), those melting between 10 and 21°C make up a middle-melting fraction (MMF), and those melting above 21°C are the high-melting fraction (HMF) (Fig. 2). Plotting the relative amounts of each fraction for the parent AMF as well as the olein and stearin fractions on a triangle diagram, as shown in Figure 3, enables the fractionation process to be monitored throughout its course. The triangle diagram clearly illustrates that during the fractionation process two phases are formed with distinctly different melting properties. Such triangle diagrams facilitate analysis of fractionation experiments because evaluation of fractionation is based not only on final products but also on intermediate products. Moreover, it helps the interested person to understand the phenomenon of fractionation in a more dynamic way.

Influence of agitation and fractionation temperature. For the first experimental central composite design, the effects of the process parameters agitation and fractionation temperature were investigated. Fractionation temperature and agitation rate ranged from 21 to 27°C and 11.5 to 14.5 rpm, respectively. Experimental combinations chosen within these ranges resulted in crystal suspensions with good filtration properties as determined in preliminary tests. The residence time for each fractionation was kept constant at 6 h.

DSC analysis was performed on both the final stearin and olein fractions. The following response parameters were derived from the DSC thermograms: %HMF, %MMF, %LMF, and offset temperature (Fig. 2). Table 1 shows the results of fitting these responses to a polynomial model (Eq. 1). We observed that only the first-order effect of fractionation temperature was significant ($P < 0.05$) on %HMF and %MMF. For both the stearin and olein fractions, higher %HMF was found at higher fractionation temperature, whereas the effect on the %MMF was the inverse. These phenomena may be enhanced at higher agitation rates as a result of better heat transfer, but significant model effects supporting this assumption were not detected, except for a significant interaction effect between fractionation temperature and agitation on %MMF of stearin (Table 1).

The effect of fractionation temperature on the %HMF of stearin can be explained as follows. First, lower crystallization rates will induce a more dense crystal network and therefore lead to less oil entrapment. Second, a more pronounced presence of higher-melting TG is expected at higher temperatures owing to solubility differences. The latter explanation is confirmed by the quadratic effect of fractionation temperature on the stearin offset temperature (Table 1). For the olein fraction, %HMF is higher at higher fractionation temperatures because fewer high-melting TG are removed under these conditions. This trend was also found when analyzing the offset temperature of olein (Table 1).

That LMF and MMF behave differently is surprising. Both fractions crystallize below the fractionation temperatures applied, and a decrease of both fractions at higher fractionation temperatures might have been expected because of the relative character of the three fraction parameters. However, no

TABLE 1
Statistical Analysis[a] of the Model Parameters (Eq. 1) Showing the Influence of Fractionation Temperature (A) and Agitation Rate (B) on the Offset Melting Point and Amount of HMF, MMF, and LMF Components of Milk Fat in Both Solid (stearin) and Liquid (olein) Fractions

Parameter[b]	*P* of *F*-test	Intercept	A	B	A^2	B^2	AB
%HMF stearin	0.0432	0.63	0.012*	N.S.	N.S.	N.S.	N.S.
%MMF stearin	0.0013	0.14	–9.514E–3**	–3.464E–6	N.S.	N.S.	7.130E–3*
%LMF stearin	0.3208						
%HMF olein	0.0002	0.079	0.041**	N.S.	N.S.	N.S.	N.S.
%MMF olein	0.0039	0.47	–0.028**	N.S.	–0.019*	N.S.	N.S.
%LMF olein	0.2881						
Offset melt stearin	0.072	47.17	0.43*	N.S.	0.45*	N.S.	N.S.
Offset melt olein	0.0005	29.34	2.10**	N.S.	N.S.	N.S.	N.S.

[a]*$P < 0.05$; **$P < 0.01$. Values with no superscript or not significant (N.S.): $P > 0.05$. If the *P*-value of the *F*-test is >0.1, entries of *t*-tests are blank; if the *P*-value of the *F*-test is <0.1, *t*-tests are mentioned.
[b]HMF, high-melting fraction; MMF, middle-melting fraction; LMF, low-melting fraction.

significant effect was observed for LMF, and this might be explained by a more pronounced effect of fractionation temperature on MMF. This phenomenon can be explained as a cocrystallization effect of middle-melting TG and high-melting TG during DSC analysis. In this way, the amount of higher-melting TG may be overestimated because the HMF represents the melting of mixed crystals from high- and middle-melting TG. This cocrystallization phenomenon was previously described by Lencki and Marangoni (13).

Also, pNMR analysis was performed on the final stearin and olein fractions to determine the SFC at different temperatures as response parameters. Table 2 shows the results of the polynomial fit of these responses to the polynomial model (Eq. 1), where, for example, the abbreviation N15 indicates the SFC measured at 15°C.

The trends were similar to those from the DSC analysis, with a significant positive first-order effect for the fractionation temperature and no significant effect for agitation rate. The trends for N35 for the stearin was an exception, with a more complex relationship being observed. Thus, by increasing the fractionation temperature, both a harder stearin and a harder olein were obtained. The harder stearin corresponds well with a higher amount of HMF as determined by DSC.

In Figure 4, the effect of fractionation temperature is visualized at a constant agitation rate. The change in melting properties followed the same pattern as seen previously. However, fractionation at 28°C resulted in a lower yield, a higher m.p. and higher %HMF in the olein compared with fractionation at 20°C. The stearin fractions had the same melting characteristics, although the stearin fractionated at 28°C had somewhat more HMF in its melting profile. Obtaining olein fractions (and by default stearin fractions) with similar melting properties but with different yields demonstrates the flexibility of the fractionation process to manufacture fractions as required to suit changing end-user requirements. For example, filtering the crystal suspension, which was held at 20°C for 3.5 h, resulted in a similar stearin and olein fraction as was obtained when filtering a crystal suspension after 6 h at 28°C. However, the yield of the former will be higher. These phenomena are probably related to cocrystallization, as described by Breitschuh and Windhab (12).

Influence of residence time and fractionation temperature. In a second central composite design set of experiments, the process parameters residence time and fractionation temperature were investigated, with the agitation rate held constant at 13 rpm. Fractionation temperature and residence time ranged from 21 to 27°C and 6 to 12 h, respectively.

In Table 3, the results of the polynomial fitting (Eq. 1) on

TABLE 2
Statistical Analysis[a] of Model Parameters (Eq. 1) Showing the Influence of Fractionation Temperature (A) and Agitation Rate (B) on Solid Fat Content Values at Different Temperatures (N5, ..., N40) for Solid (stearin) and Liquid (olein) Milk Fat Fractions

Parameter	*P* of *F*-test	Intercept	A	B	A^2	B^2	AB
N5 olein	<0.0001	50.42	1.45**	N.S.	N.S.	N.S.	N.S.
N10 olein	0.0002	40.48	1.79**	N.S.	N.S.	N.S.	N.S.
N15 olein	<0.0001	28.19	1.87**	N.S.	N.S.	N.S.	N.S.
N20 olein	0.0002	12.7	1.64**	N.S.	N.S.	N.S.	N.S.
N25 olein	<0.0001	1.32	1.34**	N.S.	N.S.	N.S.	N.S.
N20 stearin	0.0235	43.17	0.98*	N.S.	N.S.	N.S.	N.S.
N25 stearin	0.0048	31.31	1.37**	N.S.	N.S.	N.S.	N.S.
N30 stearin	0.0058	20.8	1.30**	N.S.	N.S.	N.S.	N.S.
N35 stearin	0.0016	11.8	1.20**	–0.18	0.53*	N.S.	–0.75*
N40 stearin	0.002	1.91	1.12**	N.S.	N.S.	N.S.	N.S.

[a]*$P < 0.05$; **$P < 0.01$. Without superscript or with N.S., $P > 0.05$. For abbreviations and further description of statistics see Table 1.

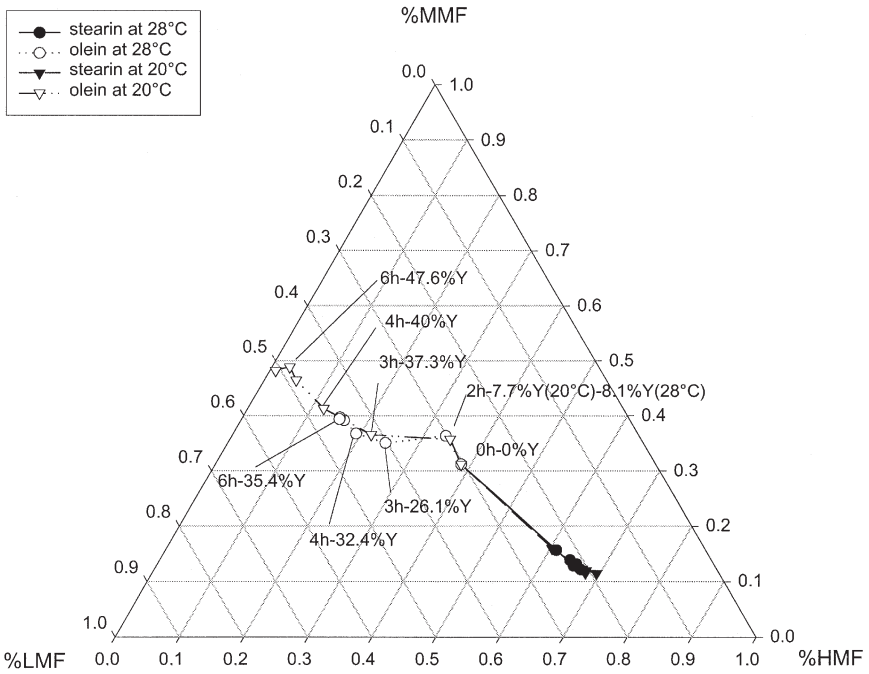

FIG. 4. Triangle diagram of the effect of fractionation temperature on melting properties. %Y = yield of stearin with an agitation rate of 13 rpm and a residence time of 6 h. For other abbreviations see Figure 2.

responses obtained by DSC analysis are outlined for the final stearin and olein fractions. Statistical analysis revealed that both residence time and fractionation temperature independently influenced melting and crystallization properties of the final stearin and olein.

By increasing the fractionation temperature, the amount of HMF in the olein was increased, whereas the amount of MMF and LMF decreased, analogous to the effects observed in the previous set of experiments. Longer residence times result in a lower %HMF in the olein at a constant fractionation temperature, illustrating that the crystallization rate is limiting for the removal of high-melting TG from the melt. Simultaneously, longer residence times result in a higher %MMF in the olein. The latter was not observed for the %LMF because of cocrystallization during DSC analysis. In practice, a balance needs to be found between olein hardness, production costs due to longer residence times, and less efficient filtration properties. From Table 3, no clear conclusions can be drawn from the effects of fractionation temperature and residence time on stearin thermal properties. This contrasts with the previous experimental set where the residence time was held constant at 6 h. Deffense (3) and Gibon and Tirtiaux (14) suggested that the influence of residence time or cooling rate is related to the selectivity of crystallization. The reason why this was not observed in the plain stearin fraction may be that either the filtration efficiency was low or the range of cooling rates applied was unsuitable. In support of our findings, Schaap and Rutten (15) also found no influence of the cooling rate on the final fractions.

Table 4 shows the results of the polynomial fitting of the responses obtained by pNMR for the final olein fraction. SFC values of the stearins were not significantly influenced by the process parameters. Olein properties were once again significantly influenced by fractionation temperature and residence

TABLE 3
Statistical Analysis[a] of the Model Parameters (Eq. 1) Showing the Influence of Fractionation Temperature (A) and Residence Time (B) on the Offset Melting Point and Amount of HMF, MMF, and LMF Components of Milk Fat in Both Solid (olein) and Liquid (olein) Fractions

Parameter	*P* of *F*-test	Intercept	A	B	A^2	B^2	AB
%HMF stearin	0.0140	0.66	–2.041E–3	N.S.	–0.020**	N.S.	N.S.
%MMF stearin	0.0136	0.13	–1.610E–4	N.S.	0.013**	N.S.	N.S.
%LMF stearin	0.2259						
%HMF olein	<0.0001	0.53	0.034**	–0.018**	N.S.	N.S.	N.S.
%MMF olein	<0.0001	0.47	–0.022**	0.015**	N.S.	N.S.	N.S.
%LMF olein	0.0179	0.47	–0.012**	N.S.	N.S.	N.S.	N.S.
Offset melt olein	0.0005	29.49	2.39**	–1.27**	1.00*	N.S.	N.S.

[a]*$P < 0.05$; **$P < 0.01$. Without superscript or with N.S., $P > 0.05$. For abbreviations and further description of statistics see Table 1.

TABLE 4
Statistical Analysis[a] of the Model Parameters (Eq. 1) showing the Influence of Fractionation Temperature (A) and Residence Time (B) on Solid Fat Content Values at Different Temperatures (N5, ..., N40) for Liquid (olein) Milk Fat Fraction

Parameter	*P* of *F*-test	Intercept	A	B	A^2	B^2	AB
N5 olein	<0.0001	50.95	1.58**	–0.48*	N.S.	N.S.	N.S.
N10 olein	<0.0001	39.95	1.70**	–0.58*	N.S.	N.S.	N.S.
N15 olein	<0.0001	27.73	2.10**	–0.54**	N.S.	N.S.	N.S.
N20 olein	<0.0001	11.22	1.71**	–0.69**	N.S.	N.S.	N.S.
N25 olein	0.0002	0.82	0.98**	–0.61**	0.38*	N.S.	N.S.

[a]*$P < 0.05$); **$P < 0.01$. Without superscript or N.S. $P > 0.05$. For further description of statistics see Table 1.

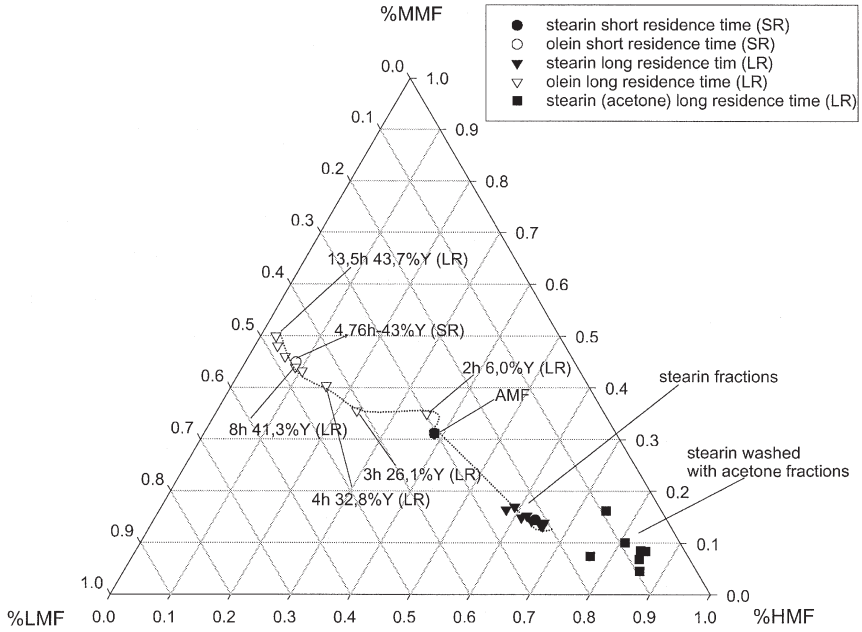

FIG. 5. Triangle diagram of the effect of residence time on the melting properties. SR, 4.5–6 h; LR, 12–13.5 h; %Y, yield of stearin at a fractionation temperature of 24°C and an agitation rate of 13 rpm. For other abbreviations see Figure 2. AMF, anhydrous milk fat.

time. By increasing temperature and decreasing residence time, a harder olein was obtained. The quadratic effect of fractionation temperature on N25 olein was similar to the effect on the offset melting temperature as determined by DSC (Table 3), illustrating a more pronounced effect at higher fractionation temperatures.

Figure 5 is a triangle diagram used to visualize two fractionation experiments at 24°C. It can be seen that similar melting properties and yields were obtained for the final stearin fractions. Probably the phenomenon of cocrystallization at shorter residence times had the same effect as a higher oil entrapment because of crystal aggregate breakdown at longer residence times. Both phenomena resulted in softer stearins. In contrast, the olein melting profile changed dramatically as a function of residence time because of incomplete removal of high-melting TG. For the experiment with a long residence time, the stearin was washed with acetone to remove part of the entrapped olein, resulting in a purer high-melting fraction.

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