Effect of Processing Conditions on Physical Properties of a Milk Fat Model System: Rheology

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ABSTRACT: The effect of processing conditions on rheological behavior of three blends of 30, 40, and 50% of high-melting fraction [melting point measured as Mettler dropping point (MDP) = 47.5°C] in low-melting fraction (MDP= 16.5°C) of milk fat was studied. The effects of cooling and agitation rates, crystallization temperature, chemical composition of the blends, and time of storage on complex, storage and loss moduli were investigated by dynamic mechanical analysis (DMA). Compression tests were performed on samples using frequency values within the linear viscoelastic range (1 to 10 Hz). Loss modulus was, on average, 10 times lower than elastic modulus and was generally not affected by processing conditions. Samples showed a more solid-like behavior that was better described by storage modulus. Storage modulus varied with all processing conditions used in this study, and even for the same solid fat content, different rheological properties were found. Storage and complex modulus increased with temperature of crystallization (25 to 30°C), even though solid fat contents of samples measured after 24 h at 10°C were the same. Moduli were higher for samples crystallized at slow cooling rate, decreased with agitation rate, and were lower for the 30–70% blend at all processing conditions used. Storage moduli also increased with storage time. Shear storage modulus was calculated from the DMA experimental data, and the results were in agreement with the values reported in literature for butter systems. Fractal dimensions calculated for these systems showed a significant decrease as agitation rate increased in agreement with the softening effect reported for working of butter.

Paper no. J9234 in *JAOCS 77,* 1189–1195 (November 2000).

KEY WORDS: Complex modulus, elastic modulus, loss modulus, milk fat fractions blends, processing conditions, rheology, solid fat content.

In food, an understanding of rheology is critical in optimizing product development efforts, processing methodology, and final product quality (1). The rheological behavior of plastic fats is governed by interactions between fat crystals in an aggregated three-dimensional, solid–liquid matrix (2). The liquid portion of the fat, interspersed throughout the aggregated fat network, serves as a continuous phase and, in conjunction with the solid fraction, is responsible for viscoelastic behavior (3). Of primary importance to the rheological behavior of fat are the amount of crystalline fat and the type of crystals present in the fat crystal network (4,5).

Rheological measurements of fats can be performed at low or high deformation. In the latter, the fat crystal network undergoes irreversible deformation, whereas in the former, viscoelasticity is measured below the yield point and any permanent strain remains upon complete release of stress. Materials can be linear elastic, elastoplastic, or nonlinear elastic. Linear elastic materials show a straight line through the origin for stress vs. strain curves. Elastoplastic materials show straight lines until the yield point is reached, and then permanent deformation occurs with higher stress. Nonlinear elastic materials do not show linear behavior in any range of stress. Margarine and butter, at room temperature, may behave as elastoplastic substances (1).

Many studies evaluating hardness and rheological properties of butters and spreads have been performed (6–13). A variety of techniques has been used, including penetrometry, Instron analysis, dynamic shear and stress relaxation, static and dynamic compression, and small-amplitude dynamic measurement using dynamic mechanical analysis (DMA). From DMA, fundamental rheological properties (complex viscosity, storage modulus, and loss modulus) of the product can be obtained. Other authors have studied the effect of blending butterfat and other fat systems. Fairley *et al.* (14) studied the mechanical properties of tripalmitin/butterfat mixtures as an alternative to hard butterfat fractions in some applications. Rousseau *et al.* (15) examined the rheological behavior of noninteresterified and interesterified butterfat and butterfat–canola oil blends by cone penetrometry and small-amplitude, constant-stress rheometry. Simoneau and German (16) investigated the effect of adding cocoa butter, as a model for long-chain monounsaturated triacylglycerols (TAG) with the unsaturated species in the *sn*-2 position, to milk fat fractions depleted in long-chain (TAG) on the textural properties of the fractions.

The solid fat content (SFC) of a fat mixture is usually used to predict hardness of a lipid product. However, processing conditions also influence the rheological characteristics of the final product. For example, agitation (or working) of butter causes a decrease in hardness without changing SFC (17). This is related to the number and structure of fat crystals present and to the interactions between these crystals.

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The aim of this study is to investigate the effect of processing conditions on rheological behavior of a milk fat model system. Different blends of high-melting fraction (HMF) in low-melting fraction (LMF) of milk fat were examined by DMA after crystallizing at different temperatures, different agitation and cooling rates. The effect of storage time on rheological properties was also studied.

MATERIALS AND METHODS

Starting blends. Three model systems were prepared by mixing 30, 40, and 50% of HMF with LMF of milk fat. Fractions were obtained from Grassland Dairy (Greenwood, WI). The Mettler dropping points (MDP) and TAG composition of the milk fat fractions are reported in Table 1, Reference 18, along with the MDP and TAG composition of the blends.

Crystallization procedure. Samples (500 g) were melted in a water bath at 80°C and kept at this temperature for 40 min. Melted samples were placed in a 1.0-L stainless-steel jacketed vessel with a 23-cm height and an 8.4-cm inner diameter. A mixer was attached to a Master Servodyne (Servodyne Controller, Chicago, IL) drive unit, which maintained constant motor speed. The mixer consisted of a 0.8-cm diameter and 39.2-cm length polypropylene shaft with a U-shaped blade paddle assembly for mixing highly viscous liquids. Maximum paddle diameter was 6.6 cm. A copper-constantan thermocouple, attached to an aluminum brace and positioned 1 cm from the center of the tank and 2 cm from the top of the sample, was used to determine sample temperature. Bath and sample temperatures were recorded. Samples were cooled at two rates. For the fast rate (5.5°C/min), a Lauda RC 20 (Lauda, Königshofen, Germany) water bath was set at crystallization temperatures of 25, 27.5, and 30°C. Cooling rate was calculated from the slope of the linear part of sample temperature profile. For the slow cooling rate (0.2°C/min), the set point temperature of the Lauda water bath was reduced from 80°C to the crystallization temperatures at a rate of 1°C every 5 min. Constant agitation rates of 50, 100, and 200 rpm were used. The crystallization process, in a sense, simulated an industrial process in that samples were crystallized at a selected temperature, after which the product was packaged and cooled for storage at another temperature. All the experiments were run in duplicate and results were averaged.

After 2 h, samples were almost completely crystallized to equilibrium for that temperature. Aliquots of the crystalline slurry were placed in nuclear magnetic resonance (NMR) tubes to measure SFC and in cylindrical plastic tubes for rheological analysis. Samples were kept for 24 h at 10°C before NMR and rheological analysis.

NMR. SFC of the samples were measured by pulsed NMR in a Minispec PC/120 series NMR analyzer (Bruker, Karlsruhe, Germany). Samples were run in duplicate and the values were averaged. The final SFC was determined for the same storage time and temperature as the rheological analysis.

DMA. Complex, storage, and loss moduli were measured by small-amplitude dynamic measurements using DMA in a DMA 7e PerkinElmer analyzer (PerkinElmer, Chicago, IL) with Pyris™ software for Windows. Compression experiments were performed in a parallel plate system of 10-mm diameter. Calibrations for height, force, eigendeformation (own deformation), and temperature of the DMA were performed prior to the study. The equipment calibration was checked prior to each use with a 10-mm quartz cylinder for height and a steel cylinder for eigendeformation. The semisolid fat samples were placed in cylindrical tubes of 10-mm diameter and cylinders from 5- to 8-mm height were cut. Temperature of samples was kept at 10°C. To determine the linear viscoelastic range (LVR) for these fat systems, a static force scan from 0.5 to 20 kPa was performed. The static and dynamic forces per area selected for the LVR were lower than the stress corresponding to the yield point, which was 14 kPa. A dynamic test from 0.5 to 4 kPa with an increasing rate of 0.5 kPa/min at a frequency of 1 Hz and a strain of 0.0001 was done to put the probe in touch with the sample surface. Values of moduli were obtained for a frequency scan from 1 to 10 Hz using a fixed stress value of 2.0 kPa dynamic force and 2.5 kPa static force. E'' describes the viscous behavior, E', the elastic or solid-like behavior, and E^* the general viscoelastic behavior of the materials. Results are the average of five runs.

Values of G' were calculated using Equation 1:

$$E' = 2G'\,(1 + \nu) \qquad [1]$$

where ν is the Poisson's ratio, which was 0.35 for our system.

Frequency increases at two different rates in this equipment because the kind of deformation performed does not allow using linear increase for probe oscillatory movement rate. Below 1 Hz, the values of complex (E^*), storage (E'), and loss (E'') moduli obtained did not correlate to the complex shear (G^*), storage shear (G'), and loss shear (G'') moduli reported in literature for butterfat. Above this frequency, a good correlation was obtained.

Evaluation of the fractal dimension. Fractal dimension of samples was evaluated with the method of Marangoni and Rousseau (19). The structure of a wide variety of objects can be described in terms of fractal geometry. A characteristic of fractal objects is their self-similarity, or in other words, fractal objects look the same under different magnifications, at least over a limited range of scales. The elastic constant of a colloidal aggregate (in our case, the fat crystal network in liquid oil) as a function of particle concentration (in our case, the SFC or volume fraction of crystals) is dictated by the fractal nature of the colloidal flocs. The colloidal aggregate is considered a collection of fractal flocs that are closely packed throughout the sample. Depending on the strength of the links between flocs relative to that of the flocs themselves, we can have strong-link behavior or weak-link behavior. Weak-link behavior, according to Equation 2, is observed at high particle concentrations (high SFC) or high crystal volume fraction:

$$G' = \gamma\,(\mathrm{SFC})^m \qquad [2]$$

The logarithm of the experimental values of G' and SFC were plotted against each other. The slope of the line, m, was derived by linear regression. The slope of this line is related to the fractal dimension of the crystal network, assuming a weak-link regime, by:

$$m = (d - 2)/(d - D) \qquad [3]$$

where d is the Euclidean dimension (i.e., 3) and D is the fractal dimension. The parameter γ was derived from the y intercept of the log–log plot of G' vs. SFC. This parameter has been related to the Young's modulus of the individual particles that make up the network (19).

RESULTS AND DISCUSSION

SFC of samples. Table 1 summarizes the SFC for the three blends in all processing conditions after 24 h at 10°C. The SFC depended on the composition of the blend, with SFC increasing ($P < 0.05$) with the content of HMF in the blend. Agitation and cooling rates, and crystallization temperatures had no effect on SFC for each blend.

Processing conditions. Since the storage modulus of a plastic fat is essentially a measure of its elasticity, or solid-like character, from the results in Table 1, one would expect the storage modulus (E') to be similar for all processing conditions. However, as shown in Figures 1 to 5, processing conditions had significant influence on the E' modulus values. The effect of cooling rate on the values of complex (E^*), storage (E'), and loss (E'') moduli as a function of frequency is shown in Figure 1. E'' generally was independent of frequency or showed only a very slight ascendant slope. E', however, increased with increasing frequency. For rapidly crystallized samples, the slope of E' with frequency was always lower than for slowly crystallized samples. Samples cooled at 0.2°C/min showed a more solid-like behavior (higher E' and higher slope) than those cooled at higher rate. E' was about three times E''. E^* showed the same behavior as E', which means that for these blends elastic behavior prevails over viscous behavior. The curves included in Figure 1 are the average of five runs. Standard deviation for E' varied from ±2 to ±5%. E' values at 1 Hz for different cooling rates were significantly different ($P < 0.05$).

TABLE 1
Solid Fat Content of Lipid Blends of HMF in LMF of Milk Fat Stored 24 h at 10¡C Prior to Analysis

		Agitation rate (rpm)					
		50		100		200	
		Cooling rate (°C/min)					
Blend	T_c (°C)	5.3	0.2	5.5	0.2	5.3	0.2
30–70%	25	37.5	38.0	38.2	37.0	37.6	37.2
40–60%	25	42.6	42.2	42.3	42.0	42.9	41.2
50–50%	25	47.0	46.4	46.9	46.8	46.8	47.0
50–50%	27.5	47.0	46.9	46.7	47.4	46.7	47.0
50–50%	30	47.3	47.1	46.8	46.1	47.1	47.1

T_c, crystallization temperature; HMF, high-melting fraction; LMF, low-melting fraction. Standard deviations for these values were lower than 1%.

Figure 2 shows the effect of agitation rate on the values of E^*, E', and E'' for rapidly (A) and slowly (B) crystallized blends during a frequency scan. Agitation produced a softening which resulted in a marked decrease in all moduli; increased agitation also produced a decrease in the slope of moduli vs. time curves. E' was again more than three times E''. E^* had the same behavior as E', showing again that elastic behavior predominated over viscous behavior in these systems. For both cooling rates, the decrease of all moduli was not linear with agitation rate. There was a larger decrease between 50 and 100 rpm than between 100 and 200 rpm, and this decrease was more noticeable at fast cooling rate for all samples.

Figure 3 shows the effect of composition on rheological behavior of blends. As expected based on the SFC values shown in Table 1, E' and E^* were the lowest for the 30–70% blend and the highest for the 50–50% blend. However, slopes of E' and E^* vs. time curves were very similar for the three blends crystallized under the same processing conditions, showing that these blends behaved in the same way despite their different solid content. Values of E^* showed that the ratio of elastic to viscous behavior was similar for the three blends. In this example, E'' showed a slightly ascendant slope with frequency. This behavior was not always found. Generally, E'' values were independent of frequency in the 1 to 10 Hz interval used.

Figure 4 shows the effect of crystallization temperature on moduli values. Table 1 shows that for the 50-50% blend, SFC values after 24 h at 10°C were the same regardless of the crys-

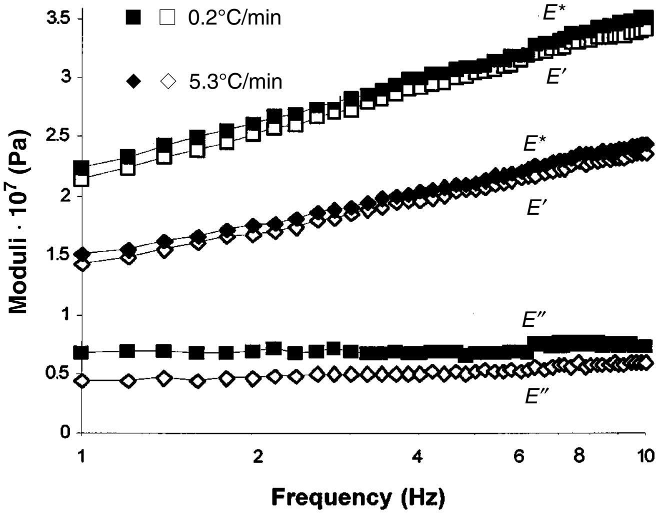

FIG. 1. Effect of cooling rate on storage (E', open symbols), loss (E''), and complex (E^*, filled symbols) modulus values measured for a 50–50% high-melting fraction (HMF) in low-melting fraction (LMF) of milk fat, crystallized at 25°C at 50 rpm, during a frequency scan from 1 to 10 Hz. Cooling rate values were 0.2 and 5.3°C/min. Samples were kept 24 h at 10°C prior to analysis. Data are average of 5 runs. Standard deviation for E' varied from ±2 to ±5%.

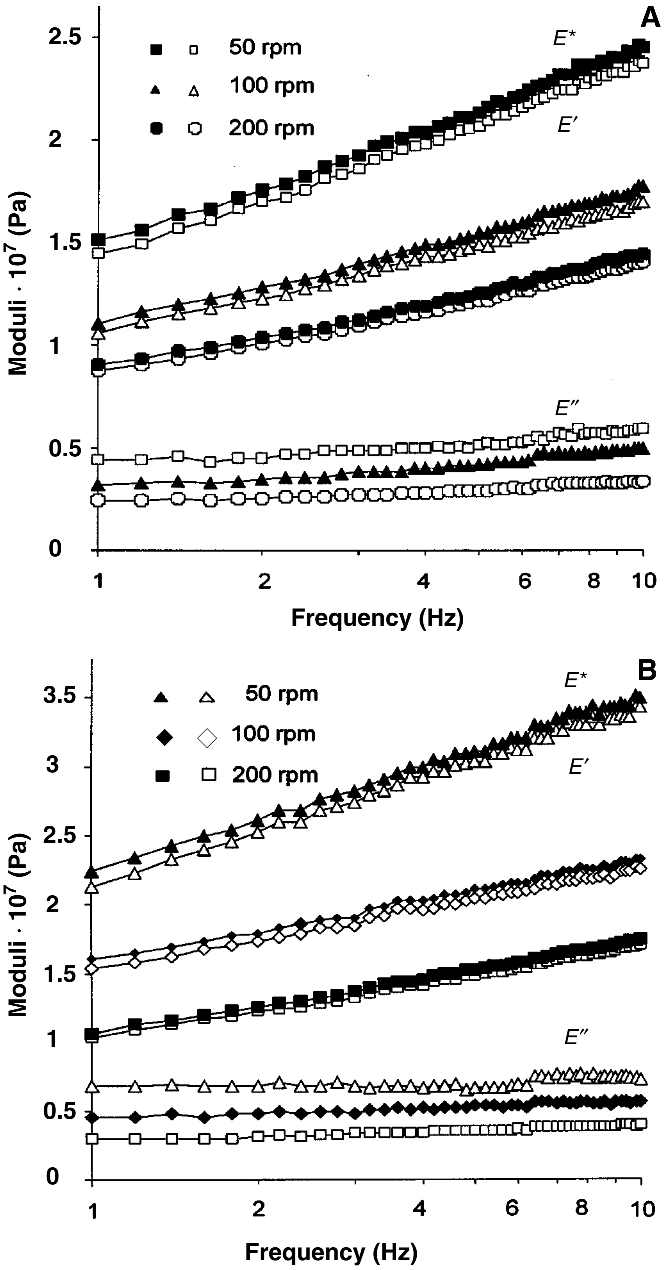

FIG. 2. Effect of agitation rate on storage (*E′*, open symbols), loss (*E″*), and complex (*E**—filled symbols) modulus values measured for a 50–50% HMF in LMF of milk fat, crystallized at 25°C at (A) 5.3°C/min and (B) 0.2°C/min, during a frequency scan from 1 to 10 Hz. Agitation rates were 50, 100, and 200 rpm in both cases. Samples were kept 24 h at 10°C prior to analysis. Data are average of five runs. Standard deviation for *E′* varied from ±2 to ±5%. For abbreviations see Figure 1.

tallization temperature, and for all agitation and cooling rates used. However, *E′* and *E** were higher for higher crystallization temperatures. Crystal size and structure were different for the different crystallization temperatures and thus differences in rheological behavior were found for the same SFC. Again, *E′* was about three times higher than *E″*, and the total viscoelastic behavior described by *E** increased with increased crystallization temperature.

Figure 5 shows the effect of storage time on the values of *E**, *E′*, and *E″* for rapidly (A) and slowly (B) crystallized blends during a frequency scan. *E″* values increased slightly with storage time in both cases, whereas *E′* and *E** values increased markedly with storage time. *E′* of slowly crystallized samples increased at least up to 3 wk, although it appears that hardening was almost complete by this time. Rapidly crystal-

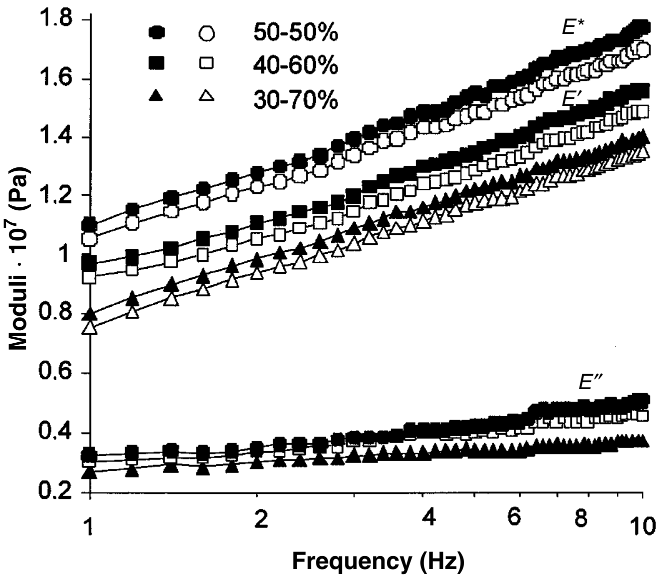

FIG. 3. Effect of composition on *E′* (open symbols), *E″*, and *E** (filled symbols) modulus values measured for blends of 30–70%, 40–60%, and 50–50% HMF in LMF of milk fat during a frequency scan from 1 to 10 Hz. Agitation rate was 100 rpm and cooling rate 5.5°C/min. Samples were kept 24 h at 10°C prior to analysis. Data are average of five runs. Standard deviation for *E′* varied from ±2 to ±5%. For abbreviations see FIgure 1.

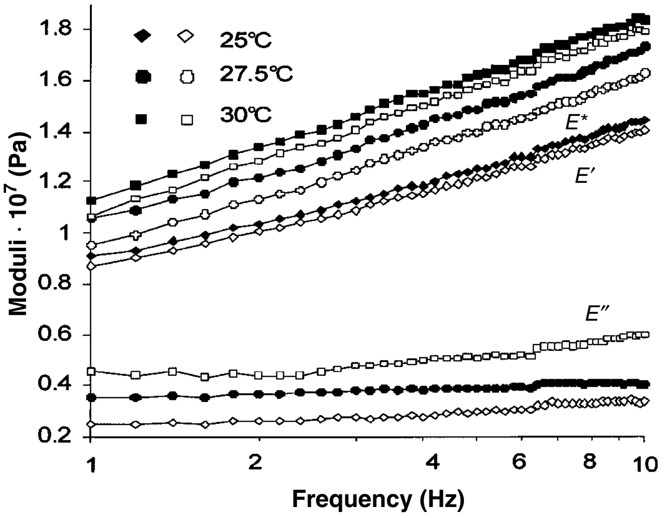

FIG. 4. Effect of temperature on storage (*E′*—open symbols), loss (*E″*), and complex (*E**—filled symbols) modulus values measured for a 50–50% HMF in LMF of milk fat crystallized at 25, 27.5, and 30°C, at 200 rpm and a cooling rate of 5.3°C/min, during a frequency scan from 1 to 10 Hz. Samples were kept 24 h at 10°C prior to analysis. Data are average of five runs. Standard deviation for *E′* varied from ±2 to ±5%.

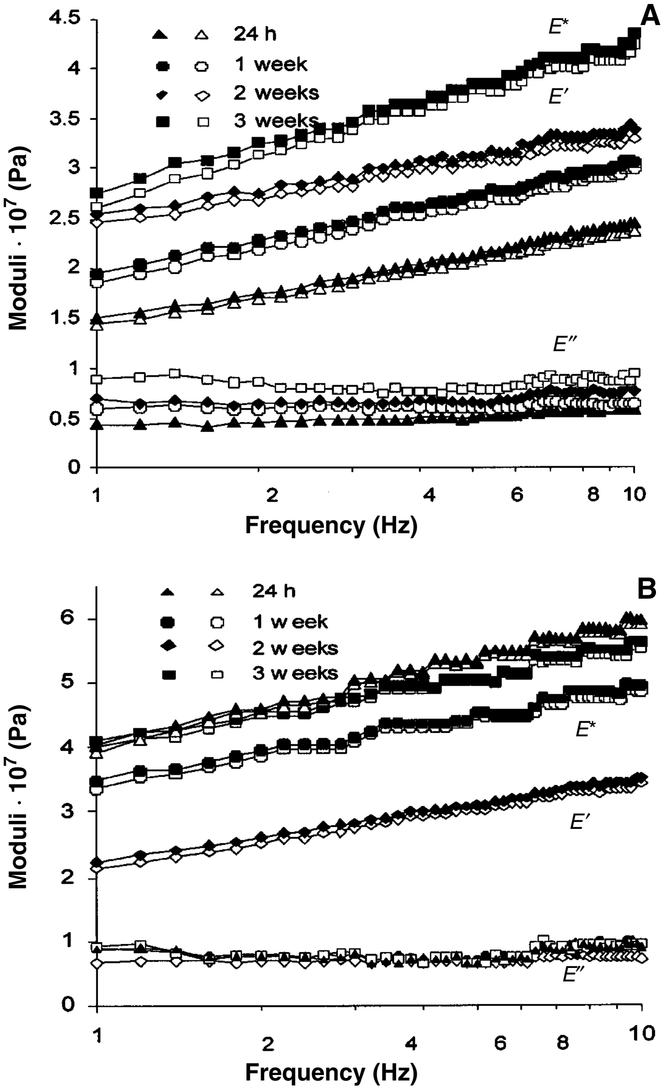

FIG. 5. Effect of storage time on storage (E'—open symbols), loss (E''), and complex (E^*—filled symbols) modulus values for a 50–50% HMF in LMF of milk fat crystallized at 25°C, 50 rpm and a cooling rate of (A) 5.3°C/min and (B) 0.2°C/min, during a frequency scan from 1 to 10 Hz. Moduli were measured after 24 h, 1 wk, 2 wk, and 3 wk at 10°C. Data are average of five runs. Standard deviation for E' varied from ±2 to ±5%. For abbreviations see Figure 1.

lized samples were continuing to increase in hardness even after 3 wk. The slopes of E' and E^* curves with time (frequency scan) increased slightly after 3 wk, as a result of hardening of the blend during storage. E' increased from $1.47 \cdot 10^7$ to $2.62 \cdot 10^7$ Pa (1 Hz) for rapidly crystallized samples (Fig. 5A) and from $2.10 \cdot 10^7$ to $3.95 \cdot 10^7$ Pa (1 Hz) for slowly crystallized samples. Rapidly crystallized samples did not reach E' values equivalent to those found for slowly crystallized samples during storage.

To summarize these effects, E' measured at a frequency of 1 Hz for the three blends crystallized under all processing conditions used in this study are shown in Table 2. E' values were higher ($P < 0.05$) for slow cooling rate, higher crystallization temperature, and slow agitation rate. They were also higher ($P < 0.05$) for the 50–50% blend for all processing conditions. For the 30–70% blend, E' values for fast cooling rate were from 6 to 16% lower than the values found for slow cooling rate, whereas for the 40–60% blend, differences in E' values between cooling rates of 8 to 24% were found. The 50–50% blend had the highest differences in E' between cooling rates, from 20 to 52%. The difference of 52% corresponded to a crystallization temperature of 25°C and an agitation rate of 50 rpm.

To compare compression experiments with shear experiments, storage shear moduli (G') were calculated from Equation 1. Shear experiments are more commonly performed on fat systems. However, slip is one of the main sources of error in viscometric measurements in semisolid foods. To avoid this problem, samples are usually glued or sandpaper is attached to both surfaces of the plates. In the DMA equipment, the probe moves in a vertical axis, so that samples do not slip and the measurements are potentially more accurate. Calculated G' values are summarized in Table 3, which were found to be on the same order of the values reported in the literature for butterfat systems (11,13–15). Thus, compression and shear measurements provide similar information on rheological properties.

Fractal analysis. Values of fractal dimension were calculated as described by Marangoni and Rousseau (19) using the data of Tables 1 and 3. Figure 6 shows the log–log plot of G' vs. SFC. Table 4 shows the calculated slope, correlation coefficient, and fractal dimension for all processing conditions. The range from 30–70% and 50–50% of HMF in LMF of milk fat was the interval of composition used in this study. HMF percentages higher than 50% were not possible, as crystallization at 25 or even 30°C was too rapid and slurries were too viscous to maintain agitation. Blends with percentage of HMF lower than 30% had a low SFC at 25°C. Although the interval of composition used in this study was narrow, the correlation found for the log–log plot was always higher than 0.98. It is an interesting fact that fractal dimensions can be calculated from the same blends (HMF in LMF of milk fat) with different SFC. According to Narine and Marangoni (5), this suggests that the spatial distribution of the fat network remains the same, regardless of the amount of solid within the network. This supposition is in agreement with the results found when the effect of composition on moduli was investigated (Fig. 3).

The fractal dimension decreased from about 2.77 to 2.49 when agitation rate increased from 50 to 200 rpm for both cooling rates. This is a substantial decrease in the fractal dimension of a colloidal aggregate, which, in our case, is the fat crystal network. This large change in the "structure" of the fat crystal aggregate network is probably responsible for the decrease in hardness due to agitation seen in Figure 2. Similar results were found by Shama and Sherman (17). The parameter γ was lowest at 50 rpm and increased for 100 and 200 rpm, according to the decrease in fractal dimension. However, the effect of cooling rate cannot be predicted by these calculations. For the same agitation rate, fractal dimension was very close for both cooling rates even though E' values were always higher for slowly crystallized samples. Differences in E' due to changes in cooling rate were larger than, or at least as large as, differences in E' due to of changes in

TABLE 2
Elastic E′ of Lipid Blends of HMF in LMF of Milk Fat Samples Measured at 1 Hz[a]

		Agitation rate (rpm)					
		50		100		200	
		Cooling rate (°C/min)					
Blend	T_c (°C)	5.3	0.2	5.5	0.2	5.3	0.2
30–70%	25	8.50×10^6	9.05×10^6	7.70×10^6	8.30×10^6	6.30×10^6	7.30×10^6
40–60%	25	1.25×10^7	1.55×10^7	8.82×10^6	9.50×10^6	7.30×10^6	8.35×10^6
50–50%	25	1.45×10^7	2.20×10^7	1.07×10^7	1.55×10^7	8.70×10^6	1.05×10^7
50–50%	27.5	1.70×10^7	2.35×10^7	1.15×10^7	1.75×10^7	9.60×10^6	1.24×10^7
50–50%	30	2.00×10^7	2.50×10^7	1.25×10^7	1.92×10^7	1.09×10^7	1.55×10^7

[a]Standard deviations varied between 2 and 5%. For other abbreviations see Table 1.

TABLE 3
Shear Modules (G′, Pa) Calculated from the Average Experimental Values of Elastic Modulus[a] of Blends of HMF in LMF of Milk Fat

		Agitation rate (rpm)					
		50		100		200	
		Cooling rate (°C/min)					
Blend	T_c (°C)	5.3	0.2	5.5	0.2	5.3	0.2
30–70%	25	3.15×10^6	3.35×10^6	2.85×10^6	3.07×10^6	2.34×10^6	2.70×10^6
40–60%	25	4.63×10^6	5.74×10^6	3.27×10^6	3.25×10^6	2.70×10^6	3.09×10^6
50–50%	25	5.37×10^6	8.15×10^6	3.96×10^6	5.74×10^6	3.22×10^6	3.89×10^6
50–50%	27.5	6.30×10^6	8.70×10^6	4.26×10^6	6.48×10^6	3.56×10^6	4.59×10^6
50–50%	30	7.41×10^6	9.26×10^6	4.63×10^6	7.12×10^6	4.04×10^6	5.74×10^6

[a]$E' = 2G'(1 + \upsilon)$ where E'' is the storage modulus measured by compression experiments, G'' storage shear modulus, and υ the Poisson's ratio equal to 0.35 in our system. For other abbreviations see Table 1.

agitation rate. Only small differences between γ were found for the different cooling rates.

Marangoni and Rousseau (19) studied the effect of interesterification in blends of butterfat–canola oil. They found a decrease in fractal dimension from 2.46 to 2.15, which was in agreement with the drastic decrease in hardness observed by cone penetrometry in the interesterified samples. Vreeker *et al.* (20) determined the value of the fractal dimension of tristearin in olive oil to be about 1.7–1.8. This value increased upon aging of the fat to $D = 2$ (20). Low fractal dimensions are indicative of low-density, open structures. Upon aging, crystallization, aggregation, and network formation processes continue, creating a denser, more compact and tightly packed structure with a higher fractal dimension.

The fractal dimension has been reported to be a better parameter for predicting rheological behavior of plastic fats than the SFC and appears to have great potential for targeting specific rheological properties of plastic fats (19). However, fractal dimensions calculated rheologically are physically signifi-

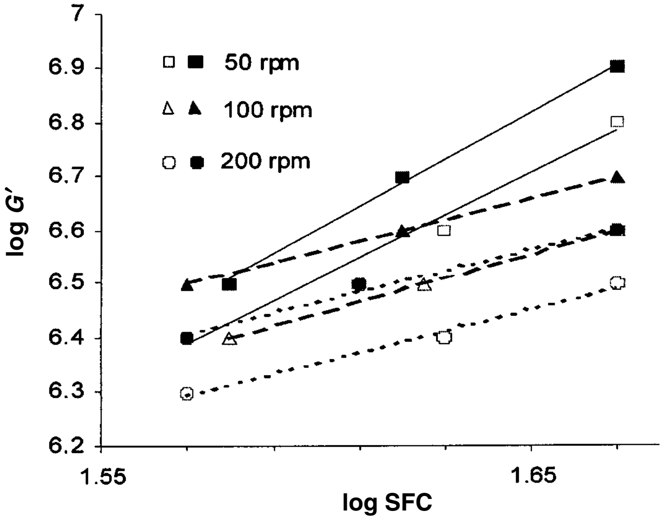

FIG. 6. Log–log plot of G' (measured at 1 Hz) vs. solid fat content for the 30–70%, 40–60%, and 50–50% blends of HMF in LMF of milk fat for all agitation and cooling rates used. Filled symbols were used to indicate slow cooling rate and open ones for fast cooling rate. For abbreviations see Figure 1.

TABLE 4
Slopes, Correlation Coefficients, Fractal Dimensions *D*, and Constant γ for the Three Blends Crystallized at 25ßC at All Agitation and Cooling Rates Used

Agitation (rpm)	Cooling rate (°C/min)	Slope	r^2	D	γ
50	0.2	4.43	0.9959	2.77	0.33
50	5.3	3.94	0.9868	2.75	1.58
100	0.2	2.22	1.0000	2.55	$2.1 \cdot 10^3$
100	5.5	2.00	1.0000	2.50	$2.5 \cdot 10^3$
200	0.2	1.97	0.9868	2.49	$9.8 \cdot 10^3$
200	5.3	1.97	0.9868	2.49	$1.2 \cdot 10^3$

cant only if the physical structure of the network is fractal in nature, i.e., self-similar on different length scales (21). The microstructure of the fat crystal network plays a key role in determining the rheological properties of plastic fats. For a deeper understanding of the rheological behavior of these milk fat systems, microstructural analysis should be done.

ACKNOWLEDGMENT

To the University of Wisconsin- Madison for its Exchange Visitor Program N° P 0105. To the National Research Council of Argentina (CONICET) for the sabbatical granted to Dr. Herrera. To Professor Alejandro Marangoni for his guidance in the development of the rheological method. To Baomin Liang for expert technical advice. This work was funded by the Wisconsin Milk Marketing Board through the Wisconsin Center for Dairy Research.

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[Received May 5, 1999; accepted July 30, 2000]