Physicochemical and Rheological Properties of Butter Made from Supercritically Fractionated Milk Fat

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ABSTRACT

A milk fat fraction enriched with high melting triglyceride was extracted by using a continuous, pilot-scale supercritical CO_2 system and recombined into butter. Compared with market butter, the high melting butter had higher contents of unsaturated long-chain fatty acids, high melting triglycerides, and β-carotene; higher intensity of yellow color; and lower cholesterol content. At similar temperatures, the high melting butter exhibited higher solid fat contents, complex viscosities, and power law consistency indices for viscoelastic parameters, indicating that its behavior is more like that of solids. The viscoelastic properties of high melting butter at 32°C were comparable with those of market butter at 22°C. The high melting triglyceride butter revealed higher emulsion stability without oiling off at 34°C. Because of its stable behavior at high temperatures, the product also offers good potential for application in bakery, chocolate, and confectionery industries in which stability of fat is a desired characteristic. Water activities were lower for high melting triglyceride butter than for market butter at similar temperatures, which should enhance microbial stability as well. Lower cholesterol and saturated fatty acid contents should also make high melting butter more attractive to consumers.

(**Key words**: physicochemical, rheology, supercritical carbon dioxide, butter).

Abbreviation key: a_w = water activity, **DSC** = differential scanning calorimeter, **G′** = storage modulus (o = intercept value), **G″** = loss modulus (o = intercept value), **HMT** = high melting triglyceride, η^* = complex viscosity (o = intercept value).

INTRODUCTION

The traditional end use of surplus milk fat has been butter. The USDA statistics (22) indicate that the annual consumption of butter has decreased from 528.6 million kg in 1985 to 407.7 million kg in 1990. The major reason for the decline in butter consumption is a growing consumer preference for reduced intake of cholesterol and saturated fats as part of a healthier life style. The availability of butter substitutes, margarines, and shortenings has also significantly contributed to this trend.

In addition to nutritional limitations, butter also has several undesirable functional attributes. Unlike many margarines and shortenings, butter fails to exhibit the desired plasticity over a wide range of temperature. At refrigeration temperatures (5 to 10°C), butter behaves like a solid and has poor spreadability. At room temperature (25°C), butter exhibits oiling off and moisture migration to the surface. In margarines and shortenings, these problems are overcome by formulations with mixtures of fats that have different physicochemical characteristics and a greater plasticity range (12).

The functional properties of butter can be improved by either physical treatment or chemical modification of the fat (3, 10, 17, 18, 20). A major limitation of the physical methods is the extent to which crystallization can be altered. This limitation led increasingly to adoption of other modification methods, such as blending with vegetable oils, addition of emulsifiers, fractionation, and reduction of fat content (3). The major obstacle to chemical modification is the strict legal requirements for butter. Butter is defined by Congress as a product made exclusively from milk, or cream,

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Received January 21, 1993.
Accepted July 28, 1993.

or both, with or without salt or coloring matter, and containing ≥80% milk fat by weight (19). Thus, reduction of fat or addition of oils or emulsifiers to improve the textural characteristics of butter invokes issues concerning standard of identity. Fractionation of milk fat appears to be the best alternative for producing modified milk fat that can be converted into butter of improved functional and nutritional value.

The separation of milk fat into fractions that are considerably different from one another in composition and physical properties offers attractive possibilities for enhancing the utility of milk fat (6). Fractionation with supercritical fluid offers new opportunities in this regard (7). A high melting triglyceride (HMT) fraction of milk fat obtained by processing milk fat with supercritical CO_2 has been found to have several desirable characteristics, such as lower cholesterol and saturated fatty acid contents and higher carotene content (7).

In addition, little literature is available on the fundamental rheological characterization of butter, modified butters, or both, because the problem of slippage is difficult to eliminate because of the greasy nature of the product. Empirical techniques, such as penetrometry, sectilometry, and extrusion have been used in the past (10, 12, 20). In empirical tests, the conditions of stress and strain existing in the sample are not known, and therefore the results cannot be expressed in terms of fundamental rheological parameters, such as viscosity and viscoelastic moduli (11, 25). Thus, dynamic mechanical analysis, a fundamental technique, was used in the present study for probing the internal structure of the butter samples via small deformation measurements.

The objectives of the present study were 1) to prepare butter from the HMT fraction of milk fat obtained by the supercritical fluid extraction process, 2) to compare the physicochemical properties of the HMT butter with those of market butter, and 3) to correlate the melting characteristics of these butter samples with their chemical composition and mechanical spectra.

MATERIALS AND METHODS

Anhydrous Milk Fat

Fresh winter market butter was purchased from the Cornell Dairy Store (Ithaca, NY), melted at 60°C, decanted, and filtered through Whatman number 1 filter paper (Arthur H. Thomas, Philadelphia, PA) under vacuum to remove protein and other materials (Figure 1). Anhydrous milk fat thus obtained was used for fractionation. Part of the same lot of market butter was used as the control.

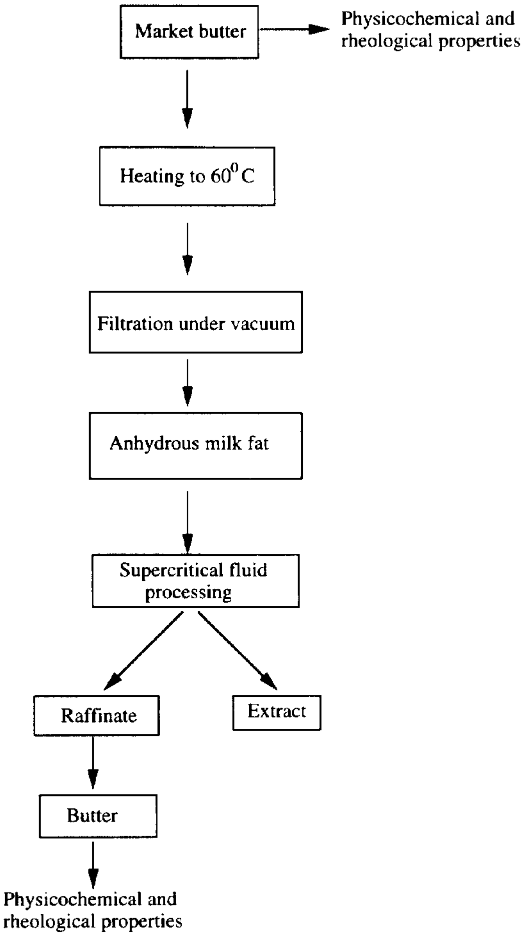

Figure 1. Flow diagram for preparation of anhydrous milk fat and its fractionation.

Supercritical Fluid Fractionation of Milk Fat

Fractionation of anhydrous milk fat into two fractions, raffinate and extract, was done on a continuous, pilot-scale supercritical CO_2 system using the process developed by Bhaskar et al. (7). The extraction was at 24.1 MPa and 40°C. The extract yield (grams of extract per gram of milk fat) was 78 g/100 g,

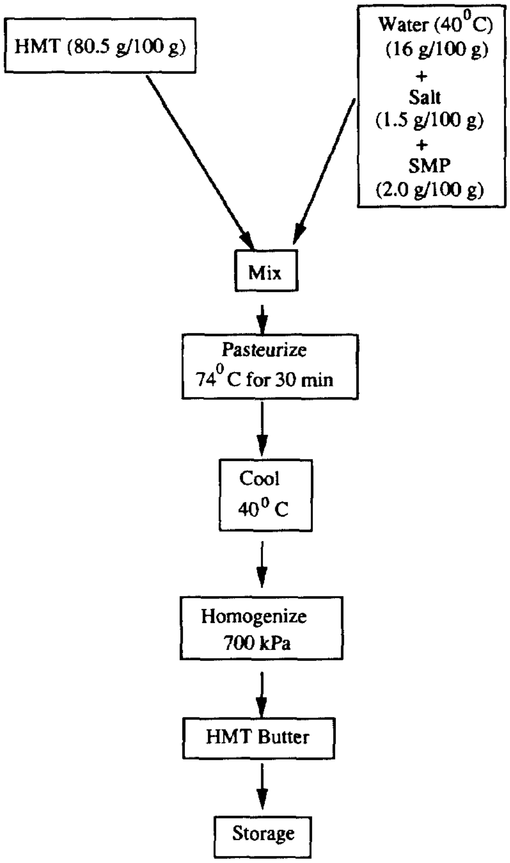

Figure 2. Flow diagram for making butter from the high melting triglyceride (HMT) fraction of milk fat.

and the raffinate yield was 21 g/100 g. The extract loading (grams of milk fat per 100 g of CO_2) was 1.27, and the solvent to feed ratio was 62. Raffinate, the fraction richer in HMT, was converted into butter.

Butter Making

Figure 2 shows the flow diagram for butter making from the HMT fraction. The HMT fraction (80.5 g/100 g) was mixed with a solution of water (16 g/100 g), salt (1.5 g/100 g), and NDM (2 g/100 g); pasteurized; and formed into butter using a single-stage homogenizer operated at 700 kPa. The homogenized samples were rapidly cooled to test temperatures and stored overnight for temperature equilibrium. Physicochemical and rheological properties of the market butter (used for fractionation) and the HMT butter were studied. Because the source of milk fat was the same, the effect of variation in feed and season was eliminated.

Chemical Analysis of Butter Fatty Acids, Triglycerides, and Cholesterol

Samples were analyzed on a gas chromatograph fitted with a flame ionization detector (HP 5890; Hewlett Packard Co., Avondale, PA). The carrier gas was helium at 1.5 ml/min.

Triglycerides were saponified into free fatty acids, followed by their subsequent derivitization of fatty acids into methyl esters (2). These methyl esters were then analyzed using a capillary column 30 m × .25 mm (Durabond-225; J&W Scientific Co., Folsom, CA).

Cholesterol was analyzed by the method of Lynch and Barbano (14), but modified to form a trimethylsillyl ether derivative. The analysis was performed using a capillary column coated with SE-30 (Chrompack Co., Middelburg, The Netherlands).

Triglycerides were analyzed by a modified method of Amer et al. (1). The analysis was performed using a capillary glass column 30 m × .25 mm (Durabond-5; J&W Scientific Co.).

Thermal Analysis

Thermal analysis of the butter samples was performed using a differential scanning calorimeter (DSC) (Model DSC-1; Perkin Elmer, Norwalk, CT) according to the procedure described by Norris et al. (16). Solid fat content at any temperature was given by the ratio of the partial area above that temperature to the total area under the DSC curve.

Carotene Content

Carotenoids were determined as β-carotene using AOAC method 938.04 (4).

Color Measurement

The color of the butter samples was measured using a Macbeth color eye spectrophotometer (Model 2020; Kollmorgen Instruments Corporation, Newberg, NY) using the Hunter scale. The Hunter L, a, and b values, which indicate the darkness, the red-

TABLE 1. Fatty acid, triglyceride, and cholesterol distribution of high melting triglycerides (HMT) and market butter samples.

Components	Butter Market	 HMT
Fatty acids	(g/100 g)	
$C_{4:0}$–$C_{8:0}$	6.76	1.22
$C_{10:0}$–$C_{12:0}$	4.69	1.95
$C_{14:0}$–$C_{18:3}$	88.55	96.83
Unsaturated (U)	32.05	41.57
Saturated (S)	56.50	55.26
U:S	.57	.75
Triglycerides		
C_{24}–C_{34}	16.72	Trace
C_{36}–C_{40}	50.85	17.07
C_{42}–C_{54}	32.93	82.93
Cholesterol, mg/100 g	240.6	117.6

ness, and the yellowness, respectively, of samples were determined.

Water Activity Measurement

Water activity (a_w) of the butter samples was measured using a commercially available a_w measuring system (Model CX-2; Decagon Devices, Pullman, WA).

Dynamic Mechanical Analysis

Small amplitude oscillatory measurements were performed on a Bohlin VOR Rheometer (Bohlin Instruments Inc., Cranbury, NJ) using a 90-g.cm torsion bar and parallel plate geometry (15-mm plate diameter and 4-mm plate gap). Four test temperatures were used in the range of 12 to 32°C. The complex viscosity (η^*), the storage modulus (G′), and the loss modulus (G″) were determined for frequencies in the range of .1 to 10 s^{-1} at strains <.1%, for which the viscoelastic properties were linear.

From the flow curves of η^* versus frequency obtained at different temperatures, master curves were obtained by shifting curves to a reference temperature (22°C) using the method of reduced variables (11).

RESULTS AND DISCUSSION

Chemical Composition

The distribution of fatty acids, triglycerides, and cholesterol in both market and HMT butters are shown in Table 1. Compared with market butter, HMT butter showed the following changes: 1) the short-chain (C_4 to C_8) fatty acids content decreased from 6.76 to 1.22%; 2) the medium-chain (C_{10} to C_{12}) fatty acid content decreased from 4.69 to 1.95%; 3) the long-chain (C_{14} to C_{18}) fatty acid content increased from 88.55 to 96.83%; 4) the unsaturated to saturated fatty acids ratio increased from .57 to .75; 5) the low melting and medium melting triglyceride concentration decreased, but the HMT concentration increased; and 6) the cholesterol content of HMT decreased by 51 g/100 g. These results indicate that the HMT butter offers the advantages of lower cholesterol and saturated fat contents. However, the contents of C_8 and C_{10}, the beneficial fatty acids, were also reduced. Also, during fractionation, the flavoring components were concentrated in the extract. Consequently, the HMT butter made from raffinate lacked the natural butter flavor, which must then be reincorporated into the product.

Carotene Content and Color Measurement

Carotene content and color values for market and HMT butter are shown in Table 2. An

TABLE 2. Comparison of color and carotenoid content of high melting triglyceride (HMT) and market butter samples.

Butter sample	Carotenoid[1]	Color[2] L	 a	 b
	(IU/100 g)			
Market	314	88.67	−1.07	24.12
HMT	768	84.08	−1.95	30.85
Change, %	+145	−5.17	−81.69	+27.89

[1] Determined as β-carotene.

[2] Hunter L, a, and b values indicate the darkness, the redness, and the yellowness, respectively, of samples.

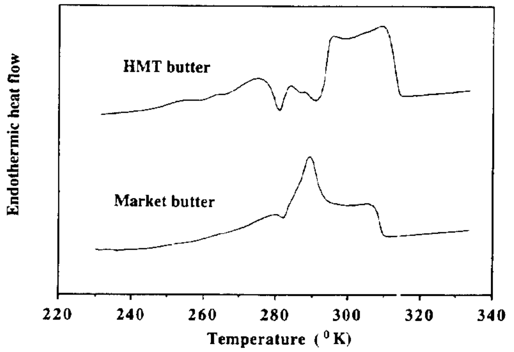

Figure 3. Melting thermograms of high melting triglyceride (HMT) and market butter samples obtained by differential scanning calorimetry.

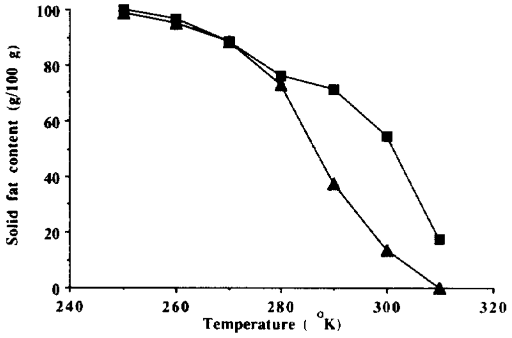

Figure 4. Solid fat contents of high melting triglyceride (HMT, ■) and market butter (▲) samples at different temperatures.

increase of 145% in β-carotene content was observed. The HMT butter showed higher b values (yellowness) than market butter.

Thermal Analysis

Thermal analysis was conducted to study the structural transitions in butter with temperature variations. Melting thermograms for HMT and market butter are displayed in Figure 3. Melting behavior of HMT butter was different from that of market butter. Samples of both butters showed three melting zones in melting thermograms: 1) a minor peak representing a low melting zone, 2) a major peak representing an intermediate melting zone, and 3) a broad shoulder displaying a high melting zone. Compared with market butter, HMT butter showed a larger area under the high melting peak in the temperature range of 291 to 315°K. This result is partly because of the higher heat of fusion of the HMT and partly because of the cocrystallization of the HMT with the low melting triglycerides to form a semistable mixture (21).

The relationship between solid fat content and temperature is shown in Figure 4. Up to 280°K, samples of both HMT and market butters exhibited similar melting behavior. However, at higher temperatures, HMT butter had a higher solid fat content than did market butter. The HMT butter revealed maximum melting (37%) from 300 to 310°K. However, market butter showed a significant drop in solid fat content (36%) from 280 to 290°K. Observations made by Vasic and DeMan (23) for market butter were similar. The difference in the melting behavior is mainly attributed to the variation in chemical composition of the butter samples. The HMT butter, being richer in HMT, displayed greater melting in the higher temperature range than did market butter.

In order to study the emulsion stability of the butters at higher temperatures, the melting behavior of HMT and market butters were visually compared at 34°C, held for 0 and 1 h (Figure 5). A higher temperature of 34°C was selected to accelerate the melting rate of fat crystals to reduce the time frame of the experiment. Market butter showed thermal sagging, but HMT butter exhibited stable behavior with no phase separation or oiling off and no moisture migration to the surface after 1 h.

a_W Measurement

The a_w for HMT and market butter are shown in Figure 6. The a_w decreased with increased temperature for HMT and market butter samples, which could be due to the melting of fat crystals and a corresponding increase in the concentration in liquid fat relative to water at the surface. Compared with market butter, a_w values were lower for HMT butter at similar temperatures, indicating less surface moisture migration. Probably, more crystalline fat inhibited the growth of larger water droplets and their subsequent rise to the surface. Lower a_w values should minimize the surface growth of yeasts and molds, a com-

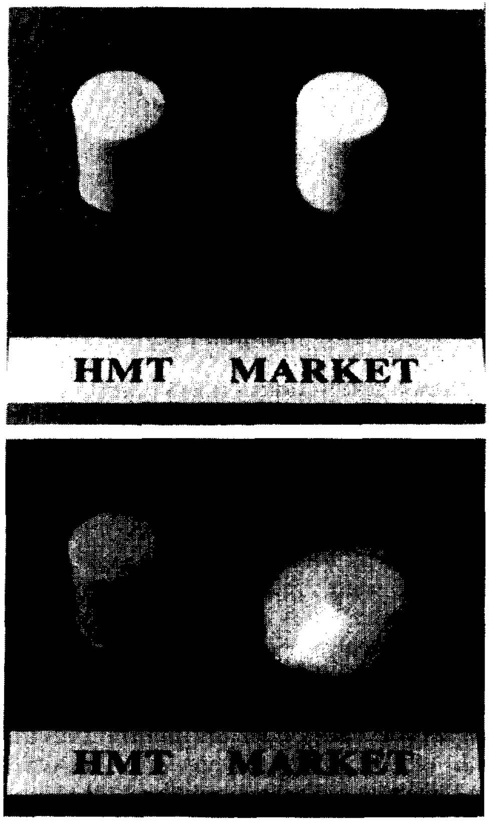

Figure 5. Comparison of melting behavior of high melting triglyceride (HMT) and market butter samples at 34°C after storage for 0 (top) and 1 h (bottom).

monly observed problem in market butter samples stored at ambient temperatures.

Dynamic Mechanical Analysis

In the present study, dynamic mechanical analysis was used to compare the rheological behavior of butter samples. Results of a representative frequency sweep profile of HMT butter sample at 32°C and market butter at 22°C are shown in Figure 7. The η^* values decreased steeply as the frequency increased, but G' and G'' showed relatively less frequency dependence. The G' values increased, but the G'' values decreased as the frequency increased. The G' values were greater than G'' values at any given frequency, indicating behavior essentially like that of solids. The frequency sweep profile for HMT butter at 32°C was similar to that for market butter at 22°C, possibly reflecting differences in solid fat content at the same temperature for the two samples.

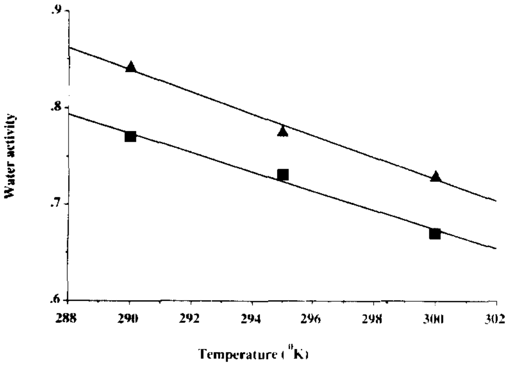

Figure 6. Water activity values of high melting triglyceride (HMT, ■) and market butter (▲) samples at different temperatures.

Because temperature markedly affects the consistency of butter, frequency sweep profiles were determined over the range of temperatures likely to be encountered in the storage and use of butter. Power law indices relating each of the rheological variable (G', G'', and η^*) to frequency (f) were determined using the following relationships (15):

$$G' = G_0' f^{n'}, \quad [1]$$

$$G'' = G_0'' f^{n''}, \quad [2]$$

and

$$\eta^* = \eta_0^* f^{n^*}. \quad [3]$$

The intercept values G_0', G_0'', and η_0^* represent the values of G', G'' and η^*, respectively, at a frequency of 1 Hz. The G_0' values increased as temperature decreased, indicating greater elastic components at lower temperatures. Trends were similar for G_0'' and η_0^* values, indicating higher loss moduli and complex viscosities at lower temperatures (Table 3). These observations could be explained by the crystallization of fat at lower temperatures, which resulted in behavior more like that of solids. Compared with market butter, HMT butter had higher

TABLE 3. Power law parameters for dynamic mechanical spectrometry frequency sweep for high melting triglyceride (HMT) and market butter samples at different temperatures.

	Butter							
	HMT				Market			
Power law index	17°C	22°C	27°C	32°C	12°C	17°C	22°C	27°C
Storage modulus (G′)								
G_0' (MPa·s$^{n'}$)	16.76	7.8	2.89	.24	11.76	6.08	.42	.02
n′	.038	.049	.047	.121	.035	.074	.122	.128
Loss modulus (G″)								
G_0'' (MPa·s$^{n''}$)	1.16	.34	.21	.04	.47	.41	.06	.004
n″	−.582	−.365	−.216	−.212	−.58	−.337	−.206	−.130
Complex viscosity (η^*)								
η_0^* (MPA·s$^{n^*}$)	2.69	1.24	.46	.039	1.77	.28	.068	.003
n*	−.968	−.952	−.953	−.894	−.967	−.929	−.887	−.886

intercept values of G_0', G_0'', and η_0^* at similar temperatures, indicating greater elastic modulus, storage modulus, and complex viscosity at 1 Hz. In particular, HMT butter at 32°C showed results that were very similar to those of market butter at 22°C, indicating better temperature stability, which again was associated with greater crystallinity of fat in HMT butter because of its higher HMT content. Similar η_0^* values for market butter at 27°C have been previously reported by Bistany and Kokini (8). Flow behavior indices (n′, n″, and n*) determine the frequency dependence of G′, G″, and η^*, respectively. The n′ values increased with increasing temperatures for both butter samples, showing that G′ is a stronger function of frequency at higher temperatures. The higher n′ values with increasing temperature indicate behavior more like that of liquids of butter samples at higher temperatures. The n″ and n* values decreased with increase in temperatures, indicating reduced frequency dependence of G″ and η^* values. Negative n″ and n* indicated declining G″ and η^* values with increasing frequency.

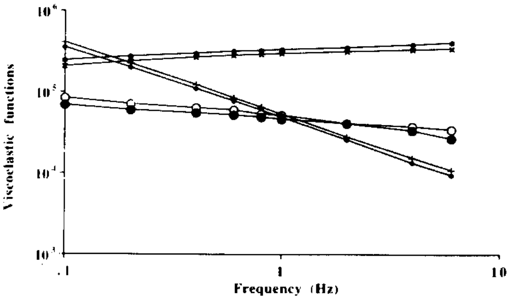

Figure 7. Dynamic mechanical spectrometry frequency sweep profiles for high melting triglyceride (HMT) butter sample at 32°C and market butter at 22°C. ♦ = Complex viscosity (η^*) for HMT butter at 32°C, x = storage modulus (G′) for market butter at 32°C, o = loss modulus (G″) for HMT butter at 32°C, + = complex viscosity (η^*) for market butter sample at 22°C, • = storage modulus (G′) for market butter sample at 22°C, ● = loss modulus (G″) for market butter at 22°C.

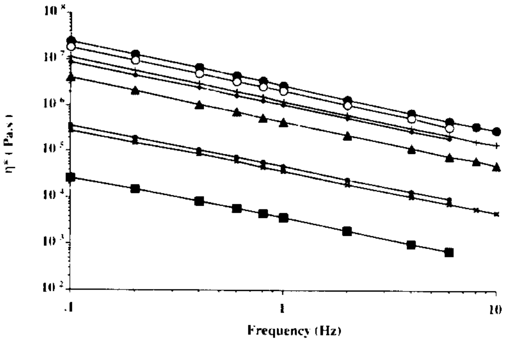

Figure 8. Complex viscosity (η^*) versus frequency (f) for high melting triglyceride (HMT) and market butter samples at different temperatures. x = HMT Butter at 32°C, ▲ = HMT butter at 27°C, + = HMT butter at 22°°C, ● = HMT butter at 17°C, ■ = market butter at 27°C, • = market butter at 22°C, ♦ = market butter at 17°C, O = market butter at 12°C.

A log-log plot of complex viscosity at different temperatures and frequencies is shown in Figure 8. All flow curves had similar slopes but different intercept values. Variation in temperature seemed to shift the curves horizontally. To evaluate the degree of this shift quantitatively, a "shift factor" (a_T) was calculated using the following relationship (11):

$$\log a_T = \log f_s - \log f = \log (f_s/f) \quad [4]$$

where f_s is the frequency of a point on the curve at a reference temperature (T_s) with a particular η^* value, and f is the frequency of a point with the same η^* on a curve at a different temperature. Using the shift factors, flow curves were superimposed using the time-temperature superposition principle (Figure 9). The master curves were obtained by plotting the log of complex viscosities at any temperature (T) against log (f/a_T), where a_T varied with temperature according to Equation [4]. Master curves indicated that HMT butter possessed higher complex viscosity than market butter samples at similar frequencies. Higher complex viscosity of HMT butter may be the reason for the improved emulsion stability of HMT butter at elevated temperatures when the market butter displayed excessive melting and oiling off.

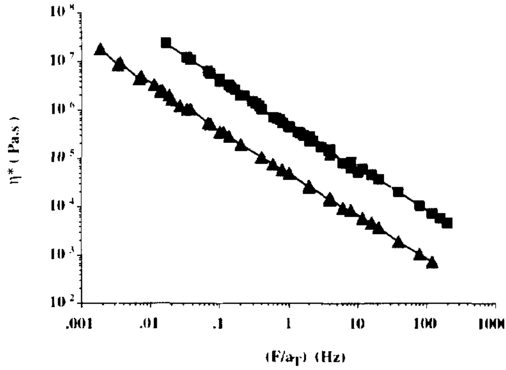

Figure 9. Master curves for complex viscosity (η^*) of market (▲) and high melting triglyceride (HMT, ■) butter samples. F is the frequency and a_T is the shift factor.

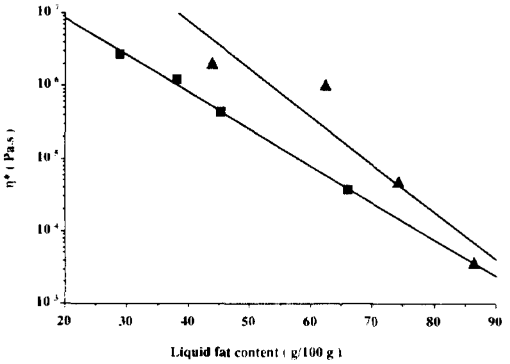

Figure 10. Complex viscosity as a function of liquid fat content for high melting triglyceride (HMT, ■) and market butter (▲) samples.

Correlation Between Thermal Analysis and Dynamic Mechanical Analysis

The relationship between liquid fat content and η^* at different temperatures was studied by thermal and dynamic mechanical analyses. Complex viscosity values at a frequency of 1 Hz from oscillatory tests at different temperatures were plotted against liquid fat content (grams/100 g) at those temperatures (Figure 10). A semilog plot was used because η^* varied exponentially with liquid fat contents. The relationship was inverse for both butter samples, indicating decreased firmness of samples with decreased crystallinity of fat. Borwankar and Bulgia reported similar observations (9). The curves for HMT and market butter had different slopes. If extrapolated, the curves seemed to converge at 100% liquid fat content, indicating that crystalline fat was the primary factor responsible for the different slopes. Using Figure 10, dynamic mechanical parameters could be predicted from the results of DSC and vice versa.

For the same liquid fat contents, η^* values for market butter were higher. This difference may be explained by the different chemical and physical states of fat in the two samples. Chemical composition of fats, fatty acid chain length, and degree of unsaturation affect the melting properties of fats (23, 24). The HMT butter has a higher content of unsaturated fats, which may account for the lower viscosity of HMT butter for the same liquid fat content.

Swern (19) reported that generally the viscosity of fats decreased as the degree of unsaturation increased. Also, the homogeneity of fats has a major influence on its consistency. Bailey (5) reported that a given proportion of crystals from a relatively heterogeneous fat gives higher firmness than the same proportion of crystalline homogeneous fat. Because the composition of HMT fat was more homogeneous than that of milk fat, resulting butter was less firm for the same proportion of liquid fat content.

CONCLUSIONS

The HMT butter exhibited modifications in nutritional and functional properties when compared with those of the market butter. The HMT butter was richer in unsaturated fatty acids and in β-carotene (by 145 g/100 g) and lower in cholesterol content (by 51 g/100 g). The solid fat contents and complex viscosities of HMT butter were higher at temperatures above refrigeration, imparting greater emulsion stability at elevated temperatures. This increased stability was reflected in higher values of η^* for HMT butter than for market butter at the same temperature. This higher viscosity improved the stability and eliminated oiling off and moisture migration to the surface for HMT butter at 34°C. The HMT butter also displayed lower a_w values, reflecting its better resistance toward microbial spoilage.

Thus, butter made from supercritically fractionated HMT shows good potential for use at ambient and higher temperatures at which the market butter normally exhibits problems such as oiling off and leakiness. Additionally, storage cost of such a product should be lower because the product is stable at room temperature and does not require refrigeration to retain its shape. Lower cholesterol and saturated fat contents of the product offer added advantages that consumers desire. This product should also find specific applications in chocolate, confectionery, and bakery industries in which stability of fat is a major concern (13).

Work is continuing in our laboratory to find additional applications for the extract, which is richer in low and medium melting triglycerides and has a higher cholesterol content.

ACKNOWLEDGMENTS

The authors thank Lorraine Rosenberry for chemical analysis and S. Rong for rheological data interpretation.

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