Restructuring Butterfat Through Blending and Chemical Interesterification. 3. Rheology

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ABSTRACT: Interesterified and noninteresterified butterfat–canola oil blends, ranging from 100% butterfat to 60:40 butterfat–canola oil (w/w) in 10% increments, were evaluated for hardness index (HI), dropping point, viscosity, and viscoelastic properties at small deformation. Both blending and chemical interesterification diminished HI in a nonlinear fashion. HI changes in interesterified blends were more pronounced than in noninteresterified blends. Dropping points yielded information on the structure of the blends. Butterfat's dropping point (DP) was 34.4°C, whereas that of interesterified butterfat was 37.0°C, which is indicative of a more structured network for the latter. DP values of blends with 60–90% butterfat (interesterified vs. noninteresterified) were not significantly different ($P < 0.05$). Interesterified blends had a higher crystallization onset temperature than did noninteresterified blends. All blends in the liquid state displayed Newtonian behavior. Oscillatory frequency sweep measurements at small amplitude showed that interesterified blends generally had lower storage moduli (G') than their noninteresterified counterparts. Both G' and G'' were frequency-dependent. Replacement of 30% butterfat by canola oil led to notable changes in small deformation measurements, whereas replacement of 20% butterfat led to big changes in large deformation measurements.
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KEY WORDS: Cone penetrometry, dropping point, hardness index, viscoelasticity, viscosity.

The rheological behavior of plastic fats is governed by interactions between fat crystals in an aggregated three-dimensional solid–liquid fat matrix (1). The prime forces that lead to network formation are Van der Waals attractive forces, because electrostatic and steric repulsion are absent (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 is the amount of crystalline fat and the type of crystals present in the fat crystal network (4).

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, is reversible, and approaches the state of the sample at rest (5,6). Single-point empirical measurement methods, such as cone penetrometry, extrusion, and simple compression, are commonly used for large deformation measurements (7), whereas more complex methods, such as controlled-stress rheometry, are generally used for small deformation measurements.

Many rheological studies have been performed on butter. Haighton (8) measured the hardness of butter and margarine by cone penetrometry. Shama and Sherman (9) studied the effect of work-softening on the viscoelastic properties of butter and margarine. Using creep compliance-time curves at low stresses, these authors determined that butter underwent smaller structural changes than margarine, thereby losing less instantaneous elasticity. Most of the lost elasticity was recovered during aging. Amer and Myhr (10) improved the low-temperature spreadability of butter by replacing 30% of the milkfat content in butter with sunflower oil. Unfortunately, the samples were too liquid at higher temperatures and could not be spread. Taylor and Norris (11) studied the physical properties of dairy spreads—spring and summer butters, high-linoleic acid butter, and an 80:20 milkfat/vegetable oil blend. At 5°C, only the butter with linoleic acid was considered spreadable. Hayakawa and deMan (12) studied the interpretation of cone penetrometry data, obtained from olive oil–tristearin mixtures, and applied several equations to best express the results obtained. Rohm and Weidinger (5) examined the rheological behavior of commercial butters at small deformations. They found that linear viscoelastic behavior was present up to a critical shear strain of 0.001 and that structure breakdown due to shear strain between 0.001 and 0.01 was fully recoverable. Rohm (6) probed the large-scale deformation properties of butter from lubricated and nonlubricated uniaxial compression, by stress relaxation in shear, and creep experiments in compression, from 5 to 20°C. He found that the force required to deform nonlubricated samples was higher than for lubricated samples. Creep compliance was dependent on compression stress at strains of ~0.01. Borwankar *et al.* (13) and Borwankar and Buliga (14) used controlled stress rheometry measurements as a function of temperature, drop points, and differential scanning calorimetry (DSC) to quantify rheological and fat crystal behavior during melting of margarine and tablespreads. Drake *et al.* (3) compared the liquid viscosity and viscoelastic properties of milkfat and milkfat–sucrose polyester blends (SPE)

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and found that, unlike the SPE blend, milkfat's storage and loss moduli were not frequency dependent. At 20°C, milkfat was more elastic than the SPE blends. Shukla *et al.* (15) examined the physicochemical and viscoelastic properties of butter manufactured from supercritical CO_2-extracted high-melting triacylglycerols (TAG) and anhydrous milkfat. A more thorough rheological examination was presented in another study (16). The authors found frequency dependence for the loss and storage moduli of the butter. With increasing frequency, the storage modulus increased whereas the loss modulus decreased.

This study examined the rheological behavior of noninteresterified and interesterified butterfat and butterfat–canola oil blends by cone penetrometry, dropping point, and small-amplitude constant-stress rheometry.

MATERIAL AND METHODS

Blend preparation and interesterification. For blend preparation and chemical interesterification methodology, please refer to Rousseau *et al.* (17). Noninteresterified is abbreviated to NIE and interesterified to IE.

Cone penetrometry (CP). The cone penetrometer (AOCS Method Cc 16-60) was used to determine the hardness index (HI) of the blends. The penetrating cone (M = 92.5 g) was placed just above the surface of the sample and released. The penetration depth in 0.1 mm increments was read on the dial after 5 s of contact.

Five replicates were performed for each sample. Penetration readings of 15–150 units were considered a suitable range for conversion to rheological properties (12).

Dropping point (DP) determination. DP were measured with the Mettler DP apparatus. The instrument, model FP83 (Mettler, Zurich, Switzerland), contains a sample cup with an opening at the bottom. Liquefied samples (80°C for 15 min) were pipetted into chilled sample holders and held at −10°C for 1 h prior to initiating measurements 12–15°C below the expected dropping point. A heating rate of 1°C/min was used.

Viscoelasticity and viscosity. A Carri-Med CSL 100 controlled stress rheometer (Mitech Corporation, Twinsburgh, OH) was used to measure viscosity and viscoelastic behavior of IE and NIE blends [100% butterfat to 60:40 butterfat/canola oil (w/w)]. This rheometer uses a stabilized low-inertia air bearing and a high-resolution digital encoder. Temperature control is maintained by a Peltier element.

Viscosity of the liquid sample was evaluated at 50°C with 4-cm parallel-plate geometry. Shear rates from 0.01 to 1000 s^{-1} were used. The temperature was maintained at 50 ± 0.1°C.

Crystallization behavior of IE and NIE blends was assessed as a function of viscosity. Temperature sweep profiles were from 60 to 5°C at a rate of 5°C/min. A 4-cm flat parallel-plate geometry was used at a constant strain of 2 s^{-1} with a 500-μm gap.

Dynamic oscillatory measurements were performed with a 2-cm parallel-plate geometry. Cylindrical samples (d = 2 cm; l = 0.5 cm) were placed in molds and maintained at 5°C for 24 h in a waterbath. To minimize wall slip, fine-grade sandpaper was attached to both the Peltier base and the parallel plate with glue. Evaluations of the linear viscoelastic range (LVR) were performed at a frequency of 1 Hz with G', η', δ, and displacement as parameters. Frequency sweeps from 0.1 to 10 Hz in the LVR at a constant displacement of 0.1 mrad were performed. G', G'', η', and tan δ were the parameters chosen for comparative purposes. Analyses were performed at 5°C.

Data were analyzed with the software provided by the manufacturer.

Sample measurements and statistical analysis. Interesterification was duplicated, and triplicate analyses were performed on blends and IE replicates for all DP, CP, and controlled-stress rheometry measurements. Statistical analysis was performed with the SAS General Linear Methods procedure (18). Differences were considered significant at $P < 0.05$.

RESULTS AND DISCUSSION

CP. CP is a rapid, yet empirical method used in the evaluation of texture (7). The HI is a simple expression of consistency and is calculated by dividing the mass (M) of the penetrating cone assembly by the depth of penetration (p) in millimeters (12):

$$HI = 0.1 \cdot M/p \text{ (g/mm)} \qquad [1]$$

During penetration of the cone assembly, the cone sinks into the fat sample until the stress exerted by the increasing contact surface of the cone is balanced by the hardness of the fat (7). In so doing, the crystal aggregate network is partially and irreversibly destroyed (19). CP is a large-deformation rheological method.

Figure 1 displays blend HI as a function of butterfat proportion (%w/w) measured by CP at 5°C. IE butterfat (IEBF) HI was 20.8% lower than that of NIE butterfat (NIEBF). The HI of NIE 90:10 blend was only 2.9% lower than NIEBF. Interesterification of the 90:10 blend (IE90:10) led to a 54.4% decline in HI compared with IEBF, while an HI difference of 62.8% was observed between NIE90:10 and IE90:10. Addition of 20% canola oil (NIE80:20 blend) produced a 51.4% drop in HI relative to butterfat and a 50% HI drop relative to NIE90:10. Interesterification of the 80:20 blend (IE80:20) led to a 75% drop in HI relative to IEBF and a 45.1% HI drop relative to NIE90:10. A 59.1% drop in HI was observed between NIE80:20 and IE80:20.

With 70% butterfat or less, the HI of both IE and NIE blends were similar. The HI of the NIE70:30 blend was 6 g/mm, whereas that of the IE70:30 blend was slightly lower at 4.5 g/mm, a 30% reduction. The HI difference became even smaller for blends that contained 60, 50, and 40% butterfat. At such low HI, the cone penetrometer was insensitive to structural differences between the IE and NIE blends.

Statistically, both the proportion of butterfat and chemical interesterification had significant effects on HI ($P < 0.0001$).

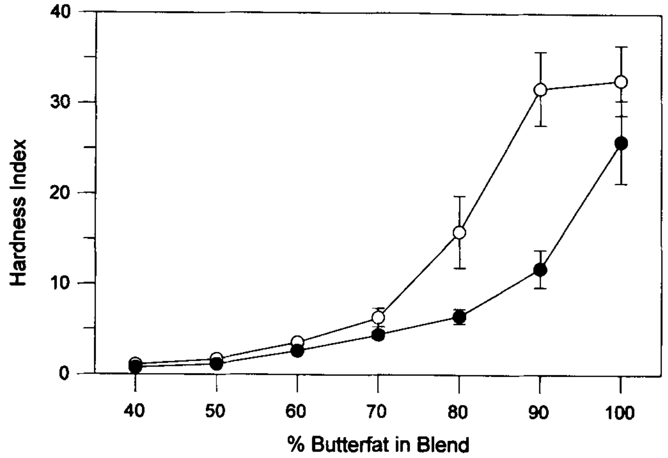

FIG. 1. Hardness index (g/mm) of noninteresterified (NIE) and interesterified (IE) butterfat–canola oil blends as a function of % butterfat (w/w) in the blend; NIE blends (O-O) and IE blends (●-●).

The HI of NIEBF and NIE90:10 were not significantly different ($P > 0.05$), whereas the HI of NIE80:20 differed significantly from all other blends. Blends with 70% or less butterfat were not significantly different from one another ($P > 0.05$). The HI of IEBF, IE90:10, and IE80:20 were all significantly different from each other ($P < 0.05$). However, IE80:20 and all IE blends containing less butterfat were not significantly different from one another ($P > 0.05$).

Blending and chemical interesterification altered the rheology of the fats *via* different mechanisms. Blending alone resulted in a dilution of the butterfat crystal network. At 5°C, butterfat is ~50% solid and 50% liquid (17). Replacement of 10% butterfat with canola oil did not substantially diminish the HI, indicating that butterfat could accommodate a larger proportion of oil at 5°C without losing structural integrity. Addition of 20% canola oil resulted in a large decrease in HI. This was interpreted as a structurally weaker network, given the lower solid fat content (SFC). Furthermore, because growth of fat crystals is slow (2) and tempering was short, interparticle interactions (e.g., sintering) were not as strong. The replacement of 30% butterfat with canola oil resulted in a low HI. Finally, with larger butterfat replacements, the HI was low. For the IE blends, the dilution effect of blending was compounded by the incorporation of unsaturated canola oil fatty acids into butterfat TAG, resulting in a lower hardness.

All CP results were obtained at 5°C. Correlation of SFC values at 5°C (17) with CP data indicated that a small difference in SFC led to large variations in rheological behavior. Thus, the role of solid fat appeared secondary when it came to CP. The small changes in SFC were not solely responsible for the substantial differences in HI observed for the IE butterfat–canola oil blends with 100 to 70% butterfat. Other mechanisms were definitely at play. As stated by Mahklouf *et al.* (20), fats with identical SFC can have vastly different rheological properties.

DP. DP for NIE and IE blends as a function of butterfat (w/w) are shown in Figure 2. Blends with 10% or less butterfat did not solidify sufficiently to yield reproducible DP. Both the proportion of butterfat and the interesterification process had significant effects on the DP of the blends ($P < 0.0001$).

According to Borwankar *et al.* (13) and Borwankar and Buliga (14), DP is not solely related to the melting point of a fat but can also reveal information on its rheological properties. In a study of margarines and table spreads, both groups found that DP was not a function of crystallinity. Instead, it correlated well with the complex viscoelastic modulus. In this study, DP was considered a rheological parameter that describes the flow that occurs at the temperature at which the fat crystal network lacks sufficient cohesion to hold oil in its matrix.

NIEBF had a DP of 34.4°C, while the IEBF value was 37.0°C. The higher DP for IEBF suggested a more structured, denser crystal network than for natural butterfat. Samples with 60–90% butterfat did not exhibit notable DP differences between NIE and IE samples, indicating that interesterification resulted in rheologically similar flow patterns for both NIE and IE samples. Samples with ≥50% canola oil displayed differences of 5–6°C between the IE and NIE samples. This suggested that the crystal network of the IE fats was weaker than that of NIE samples.

Statistically, all NIE samples were significantly different from one another ($P < 0.05$), except for NIE60:40 and NIE50:50 ($P > 0.05$). IE80:20 and IE70:30 were not different from each other; neither were IE70:30 and IE60:40 ($P > 0.05$). All other samples were significantly different from one another ($P < 0.05$). The DP of IE samples demonstrated greater change as the proportion of oil increased compared with NIE samples.

As the sample temperature is raised during a DP measurement, solid fat melts. The progressive reduction of crystalline matter means that at a certain temperature, the oil cannot be enclosed any further within the fat matrix and begins to flow out. According to deMan *et al.* (21), the DP of butter occurs at an SFC of ~2.5%. In terms of rheological properties, only a small amount of solid fat is required for butterfat to maintain a cohesive network of fat crystals and oil.

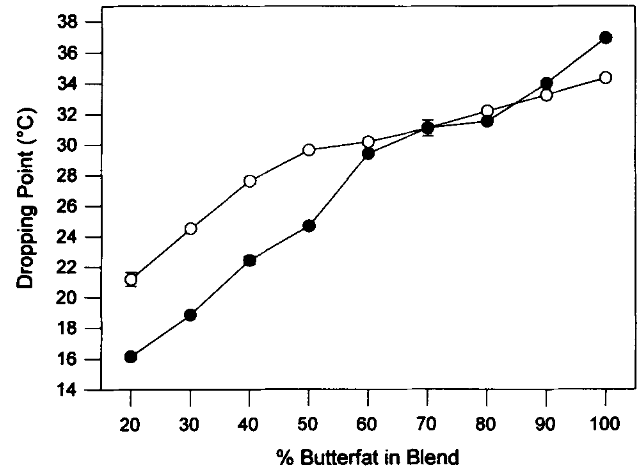

FIG. 2. Dropping point evolution (°C) of NIE and IE butterfat–canola oil blends as a function of % butterfat (w/w) in the blend; NIE blends (O-O) and IE blends (●-●). See Figure 1 for abbreviations.

Constant stress rheometry: Viscosity. Table 1 lists the viscosity of the liquid fats for blends composed of 100% butterfat to 60:40 butterfat/canola at 50°C. All fats in the liquid state exhibited Newtonian behavior (22). No discernible trend could be observed, and little difference existed between the NIE and IE blends. The only perplexing difference occurred between the NIE70:30 and IE70:30 blends. NIE70:30 had a viscosity of 23.7 mPa·s while that of IE70:30 was 29 mPa·s.

The proportion of butterfat in the blend had a significant effect on liquid viscosity ($P < 0.0001$), whereas interesterification did not ($P > 0.05$). NIE90:10 and NIE60:40 were not significantly different from each other, and neither were NIE80:20 and NIEBF. Only IE70:30 differed significantly ($P < 0.05$) from the other IE blends.

Figure 3 shows the onset of crystallization temperatures of NIE and IE blends (100 to 60% butterfat in the blend). The onset temperature (T_0) was extrapolated back to the base-line by linear regression of the dramatic increase in viscosity on the viscosity–temperature profile. Figure 4 shows the butterfat profile. At the crystallization onset temperature, the samples began to exhibit viscoelastic behavior. All IE samples had a higher crystallization onset temperature than their NIE counterparts. Interestingly, IE70:30 had a much higher crystallization onset temperature than NIE70:30, suggesting that the viscosity in the liquid probably influenced the onset of crystallization. Omitting the IE70:30 result, the difference between onset temperature of IE and NIE blends diminished with increasing oil proportion.

Both butterfat proportion and interesterification had significant effects on crystallization onset temperature ($P < 0.0001$). NIEBF and NIE90:10 were not significantly different ($P > 0.05$), nor were NIE90:10, NIE80:20, and NIE70:30. Finally, NIE80:20, NIE70:30, and NIE60:40 did not differ significantly ($P > 0.05$). For the IE blends, only IE80:20 and IE70:30 were not significantly different from all other IE blends ($P > 0.05$).

The higher crystallization onset temperatures of the IE blends are not readily explainable. A plausible explanation is that the randomization of saturated fatty acids created a greater number of potential nucleation sites in the IE blends than the number present in the NIE blends. For example, dilution of butterfat with 10% canola led to a drop in crystallization onset temperature. Interesterification of the 90:10 blend increased the onset temperature. Quite possibly, the triunsaturated (UUU) TAG present in the canola oil were restructured and now contained a saturated fatty acid (SUU). While not examined, the crystallization rate would probably have been slower for the IE blends.

TABLE 1
Viscosity (mPa·s) of Noninteresterified (NIE) and Interesterified (IE) Butterfat-Canola Oil Blends as Measured by Controlled Stress Rheometry at 50°C

Blend	NIE viscosity (mPa·s) ± SE	IE viscosity (mPa·s) ± SE
Butterfat	24.73 ± 0.08	24.21 ± 0.33
90:10	26.06 ± 0.05	25.99 ± 0.03
80:20	25.07 ± 0.27	24.29 ± 0.02
70:30	23.72 ± 0.11	28.99 ± 0.04
60:40	25.81 ± 0.04	26.09 ± 0.02

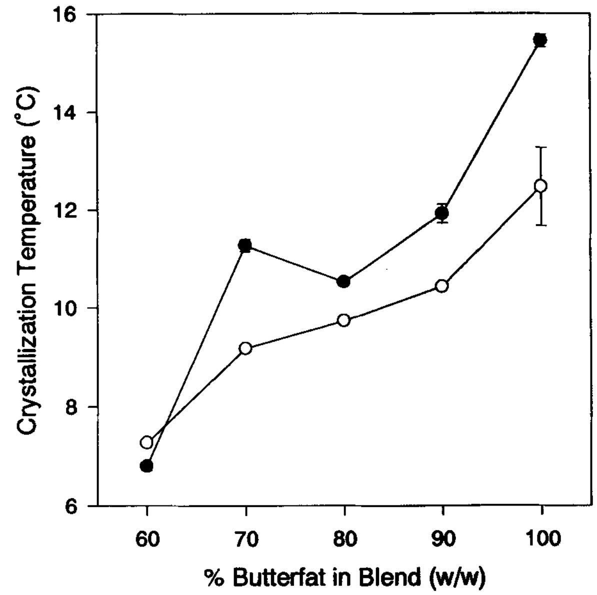

FIG. 3. Crystallization temperature (°C) as a function of viscosity of NIE and IE butterfat–canola oil blends as a function of % butterfat (w/w) in the blend; NIE blends (O-O) and IE blends (●-●). See Figure 1 for abbreviations.

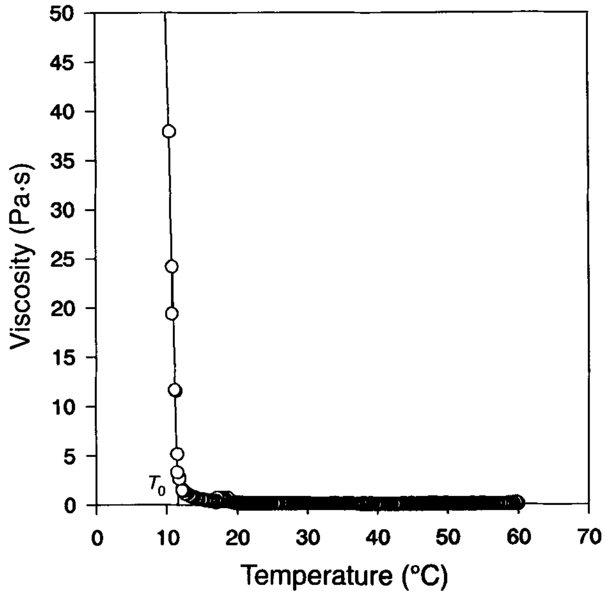

FIG. 4. NIE butterfat crystallization curve, generated by measuring viscosity as a function of temperature. Crystallization onset (T_0) was considered the temperature at which an exponential increase in viscosity was observed.

Viscoelastic properties. The parameters measured were storage modulus (G'), loss modulus (G''), dynamic viscosity (η'), and tan δ (adimensional). The storage modulus (G') is a measure of the energy stored and recovered per cycle of sinusoidal deformation and is indicative of the elastic nature of a substance, while the loss modulus (G'') is a measure of the en-

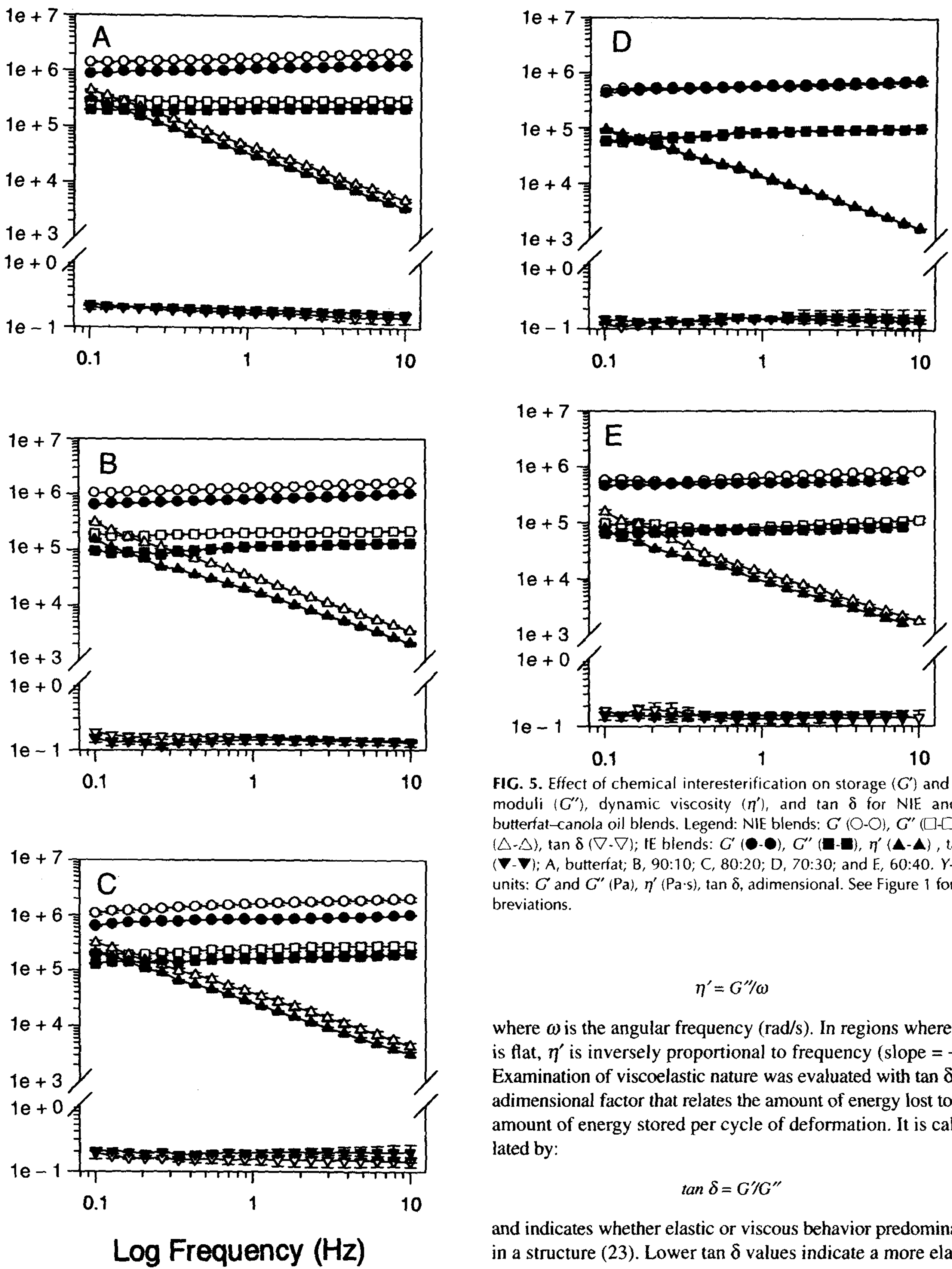

FIG. 5. Effect of chemical interesterification on storage (G') and loss moduli (G''), dynamic viscosity (η'), and tan δ for NIE and IE butterfat–canola oil blends. Legend: NIE blends: G' (○-○), G'' (□-□), η' (△-△), tan δ (▽-▽); IE blends: G' (●-●), G'' (■-■), η' (▲-▲), tan δ (▼-▼); A, butterfat; B, 90:10; C, 80:20; D, 70:30; and E, 60:40. Y-axis units: G' and G'' (Pa), η' (Pa·s), tan δ, adimensional. See Figure 1 for abbreviations.

ergy lost per cycle of sinusoidal deformation and is indicative of the viscous nature of a substance (23). Dynamic viscosity (η') is related to G'' by:

$$\eta' = G''/\omega \qquad [2]$$

where ω is the angular frequency (rad/s). In regions where G'' is flat, η' is inversely proportional to frequency (slope = −1). Examination of viscoelastic nature was evaluated with tan δ, an adimensional factor that relates the amount of energy lost to the amount of energy stored per cycle of deformation. It is calculated by:

$$\tan\delta = G'/G'' \qquad [3]$$

and indicates whether elastic or viscous behavior predominates in a structure (23). Lower tan δ values indicate a more elastic sample.

Figure 5 shows the effect of interesterification on G', G'', η', and tan δ of the blends. The G' of NIE blends was consistently higher than the G' of their IE counterparts, indicating that interesterification led to a loss of elasticity. G'' was also lower for the IE blends than their NIE counterparts. G' was frequency dependent, a phenomenon in many food systems (24) and a finding which agrees with the results of Shukla and Rizvi (16) and Rohm and Weidinger (5), who examined the viscoelastic properties of butter. According to Kokini and Plutchok (25), G' frequency dependence at high frequency is the result of interchain entanglements not having enough time to come apart within the period of one oscillation. Values of η' indicated that, with more canola oil in the blend, the frequency dependence of G'' increased. Slopes of butterfat (both IE and NIE) were very close to –1 (–0.98 and –0.96, respectively), but approached –0.86 with 30 or 40% canola oil in the blend. Values of tan δ generally decreased with increasing frequency. However, both IE and NIE blends containing larger proportions of oil (30–40%) showed little variation in tan δ as a function of frequency.

Figure 6A displays the evolution of G' and G'' as a function of the proportion of butterfat in the blends. Instead of exhibiting linear changes in G' and G'', frequency sweep spectra revealed two distinct groups. G' varied little among NIEBF, NIE90:10, and NIE80:20 and also between NIE70:30 and NIE60:40. Overall, G' dropped ~ 2.5 orders of magnitude from NIEBF to NIE60:40. These results indicate that the crystal network can maintain a rheologically cohesive structure until ~30% butterfat in the blend has been replaced with canola oil. This is in contrast to the CP data (large deformation), which indicate a large drop in relative hardness with addition of 20% canola oil.

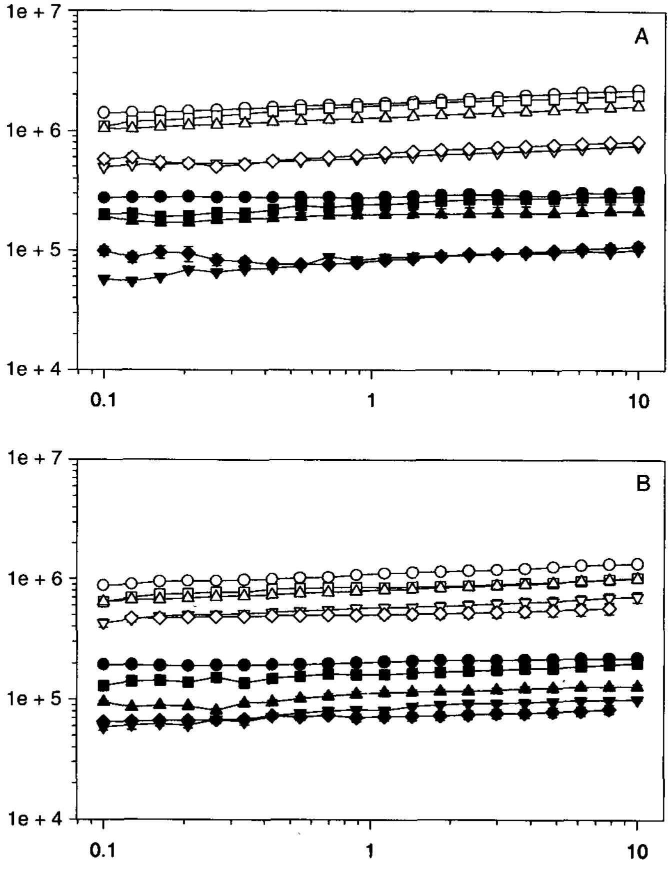

FIG. 6. Effect of blending on storage (G') and loss moduli (G'') as a function of frequency for NIE and IE butterfat–canola oil blends. A, NIE blends and B, IE blends: Butterfat—G' (○-○), G'' (●-●); 90:10—G' (□-□), G''— (■-■); 80:20—G' (△-△), G'' (▲-▲); 70:30—G' (▽-▽), G'' (▼-▼); 60:40 G' (◇-◇), G'' (◆-◆). See Figure 1 for abbreviations.

Figure 6B shows the evolution of G' and G'' for the IE blends as a function of frequency. From IEBF to IE60:40, the G' values dropped ~2 orders of magnitude. Instead of exhibiting two distinct G' groups, like the NIE blends, the G' values fell more gradually. Rohm and Weidinger (5) reported that rheological parameters of 13 Austrian butters were strongly dependent on temperature, this being related to SFC. Similarly, in this study, SFC had a profound effect on the viscoelastic characteristics of all blends. The shapes of the curves were quite similar for both IE and NIE samples, indicating that all samples exhibited similar viscoelastic behavior, but on a different scale (26).

Examination of the evolution of G'' in Figure 6 revealed that G'' diminished more than G' for both IE and NIE blends as a function of butterfat (w/w). The drop was ~ 3 orders of magnitude in both Figure 6. According to Kokini and Plutchok (25), for hydrocolloid gels (interwoven network of macromolecules), G' dominates over G'' because the network bonding forces prevent translational movement. In a plastic fat, the intertwined crystal aggregates perform a similar function, leading to increased G' over G'' values.

Interestingly, the G' values and HI correlated well ($r > 0.93$) (Fig. 7). While this may be so for only this system, it nonetheless constitutes an interesting result. According to van den Tempel (27), two types of bonds are present in fat systems—primary bonds and secondary bonds. Primary

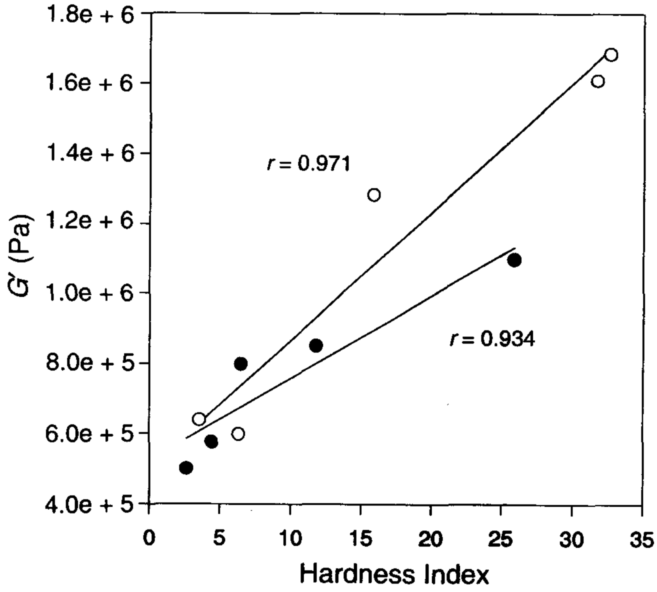

FIG. 7. Storage modulus (G') at 1 Hz vs. hardness index (g/mm) for NIE and IE butterfat–canola oil blends as a function of % butterfat (w/w) in the blend; NIE blends (○-○) and IE blends (●-●). See Figure 1 for abbreviations.

bonds result from crystals growing together and are irreversible because they do not reform after rupture. Secondary bonds, on the other hand, are due to reversible London–van der Waals forces and can reform after being ruptured (28). The former are responsible for hardness in fats, while the latter represent the forces at work during viscoelastic measurements. Based on the good correlation obtained, it appears possible to correlate hardness, a large deformation parameter (primary bonds) to G', a small deformation parameter (secondary bonds).

This study has shown that the different network forces and bonds at play in this butterfat–canola oil system depend strongly on the proportion of each component lipid. No direct correlation between SFC, polymorphic form and rheological properties could be readily established.

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