

# The Influence of Chemical Interesterification on Physicochemical Properties of Complex Fat Systems

## 1. Melting and Crystallization

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**ABSTRACT:** The effects of blending palm oil (PO) with soybean oil (SBO) and lard with canola oil, and subsequent chemical interesterification (CIE), on their melting and crystallization behavior were investigated. Lard underwent larger CIE-induced changes in triacylglycerol (TAG) composition than palm oil. Within 30 min to 1 h of CIE, changes in TAG profile appeared complete for both lard and PO. PO had a solid fat content (SFC) of ~68% at 0 C, which diminished by ~30% between 10 and 20 C. Dilution with SBO gradually lowered the initial SFC. CIE linearized the melting profile of all palm oil–soybean oil (POSBO) blends between 5 and 40 C. Lard SFC followed an entirely different trend. The melting behavior of lard and lard–canola oil (LCO) blends in the 0–40 C range was linear. CIE led to more abrupt melting for all LCO blends. Both systems displayed monotectic behavior. CIE increased the DP of POSBO blends with  $\geq 80\%$  PO in the blend and lowered that of blends with  $\leq 70\%$  PO. All CIE LCO blends had a slightly lower DP *vis- -vis* their noninteresterified counterparts. *JAOCS* 75, 1265–1271 (1998).

**KEY WORDS:** Blending, chemical interesterification, crystallization, dropping point, lard, palm oil, solid fat content, solubility.

Our present understanding of the nature of fat crystal networks is limited. Solid fat crystals and crystal agglomerates in spreads interact to form a continuous three-dimensional solid-fat network, which is ultimately responsible for the rheological properties of the plastic fat system. However, little is known about this three-dimensional network.

Factors that influence the network properties of plastic fats include (i) crystal habit, which includes crystal polymorphism, size, number, shape, and aggregation behavior; (ii) chemical composition; (iii) solid/liquid ratios; (iv) triacylglycerol (TAG)-TAG intersolubility; (v) crystallization procedure including cooling rate, degree of supercooling, mechanical working, and tempering; and (vi) the presence of non-TAG components, such as phospholipids and sterols (1–3). Linear and nonlinear interactions among some or all of these factors are also possible.

There is a growing trend in the spreadable fats industry to incorporate as much monounsaturated liquid oil as possible

into spreads for nutritional reasons. However, this has led to technical difficulties with respect to spread stability and textural properties. Another trend is the increased use of interesterification, which is a method of choice in the fat industry to manipulate spread properties (4). Reviews on interesterification have been presented elsewhere (5–7). As shown in the past with complex systems (e.g., lard, milk fat), interesterification can substantially alter the physical properties of fats.

Within this framework, the purpose of this study was to evaluate the physical, crystal, and rheological properties of Palm oil–soybean oil (POSBO) and lard–canola oil (LCO) systems after blending and chemical interesterification. Quantification of microstructure was performed by a fractal geometry approach. In the first of three parts, we examine the melting and crystallization behavior of these two systems.

### EXPERIMENTAL PROCEDURES

Neutralized, bleached, and deodorized palm oil (PO) (Jendarata Estate, Perak, Malaysia) was a gift from Dr. John deMan (University of Guelph). Commercial lard (Tenderflake, Maple Leaf Grocery Products, Toronto, Canada), which consisted of 95% natural lard and 5% hydrogenated lard, and refined soybean oil (SBO) and canola oils (CO) were obtained from a local store.

*Blend preparation.* PO and lard samples were melted at 80 C and mixed with SBO and CO, respectively. Blends of POSBO and LCO were prepared in 10% (w/w) increments from 100% lard or PO to 50% (w/w) POSBO or LCO.

Chemical interesterification (CIE) reactions were performed under vacuum (0.1 Torr) with 0.5% (w/w) sodium methylate (Sigma, St. Louis, MO) as the catalyst. Interesterification temperature was 72 C for the PO-containing samples and 80 C for the lard-containing samples. POSBO blends were interesterified for 0, 15, and 30 min and 1, 2, 3, 4, 6, and 12 h, while LCO samples were interesterified for 0, 15, and 30 min and 1, 2, 3, 4, 6, 12, and 24 h. Reaction onset was considered as the appearance of a red-brown-colored substance, usually associated with the presence of a diacylglycerol metal derivative—the active catalyst (6,8,9).

*Analysis.* Interesterified and noninteresterified (NIE) samples were analyzed for triacylglycerol (TAG) species by high-temperature gas–liquid chromatography as previously described

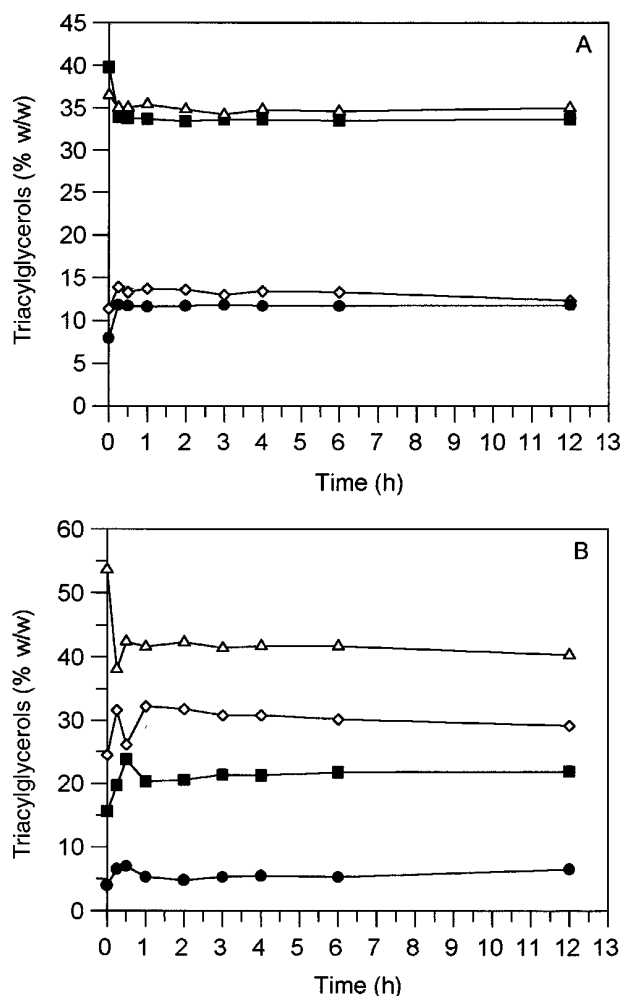
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(10). Solid fat content (SFC) of the samples was determined by pulsed-nuclear magnetic resonance as previously described (10). Dropping points of the samples were determined as previously described (11). All analyses were performed in triplicate.

Crystallization behavior of NIE and CIE samples were performed in a DuPont 1090 differential scanning calorimeter (TA Instruments, Mississauga, Ontario, Canada). Liquefied samples (8–10 mg) were loaded in aluminum pans and hermetically sealed. Samples were heated to 80°C to destroy crystal history and then cooled at 5°C/min against liquid nitrogen. An empty pan was used as the reference. Analyses were performed in triplicate. Thermograms were analyzed for onset and end of crystallization, major peak maxima temperatures (°C), and enthalpy of melting (J/g). Data analysis was performed with software provided with the calorimeter.

## RESULTS AND DISCUSSION

*Effect of interesterification duration.* Figure 1A shows the changes in PO TAG species (carbon number) as a function of



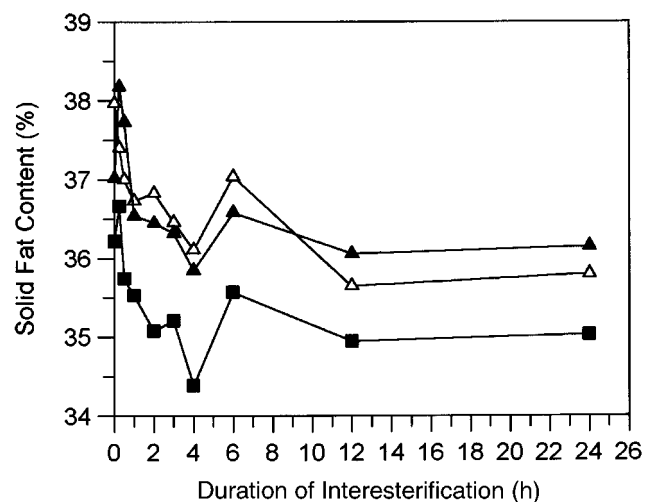
**FIG. 1.** Effect of chemical interesterification on the relative triacylglycerol (TAG) proportions (% w/w) as a function of interesterification duration. (A) Palm oil; (B) lard, 48 carbons (●-●); 50 carbons (■-■); 52 carbons (△-△); 54 carbons (◇-◇).

CIE duration. PO predominantly consisted of 50- (~40%) and 52- (~36%) carbon (excluding glycerol) TAG species with lesser amounts of 54- (~11%) and 48- (~8%) carbon TAG species, in agreement with former work (12). CIE for 30 min led to 6 and 1% decreases in the 50- and 52-carbon TAG species, respectively, and increases of 3 and 4% for the 54- and 48-carbon species, respectively. Subsequent CIE did not noticeably alter the PO TAG composition further. Wiedermann (13) reported that PO was not much altered by CIE.

Lard consisted primarily of 52- (~54%) carbon TAG species with lesser amounts of 48- (4%), 50- (~16%) and 54- (~25%) carbon TAG species. Interesterification substantially altered lard TAG composition (Fig. 1B). Lard CIE for 1 h and longer altered the distribution as follows: 48, +1%; 50, +5%, 52, -12%, and 54, +6%. These changes in TAG composition were smaller than those reported in other studies (14). Changes in SFC as a function of interesterification duration for lard are shown in Figure 2. Even though TAG modifications stopped after 1 h, the SFC kept dropping for 3–4 h. Similar qualitative trends were observed for samples that were crystallized for three different time periods. This result highlights the importance of measuring several parameters during interesterification to judge whether the desired end point of the reaction has been reached. It is curious that the opposite effect was observed for butterfat (10). In that material, the SFC stopped changing after 30 min to 1 h of interesterification, while TAG profiles kept on changing for several hours.

The importance of understanding compositional modifications through interesterification is important because generation of new TAG species can profoundly affect the physical properties of fats (15).

*SFC.* The SFC profiles as function of temperature for NIE and CIE blends of POSBO are shown in Figures 3A and 3B, respectively, while those for NIE and CIE blends of LCO are shown in Figures 3C and 3D, respectively. The SFC profiles of all blends were significantly different from each other ( $P < 0.0001$ ).



**FIG. 2.** Effect of chemical interesterification on the solid fat content (%) of lard as a function of interesterification duration. Lard samples were crystallized for 22 h (■-■); 68 h (△-△); and 167 h (▲-▲).

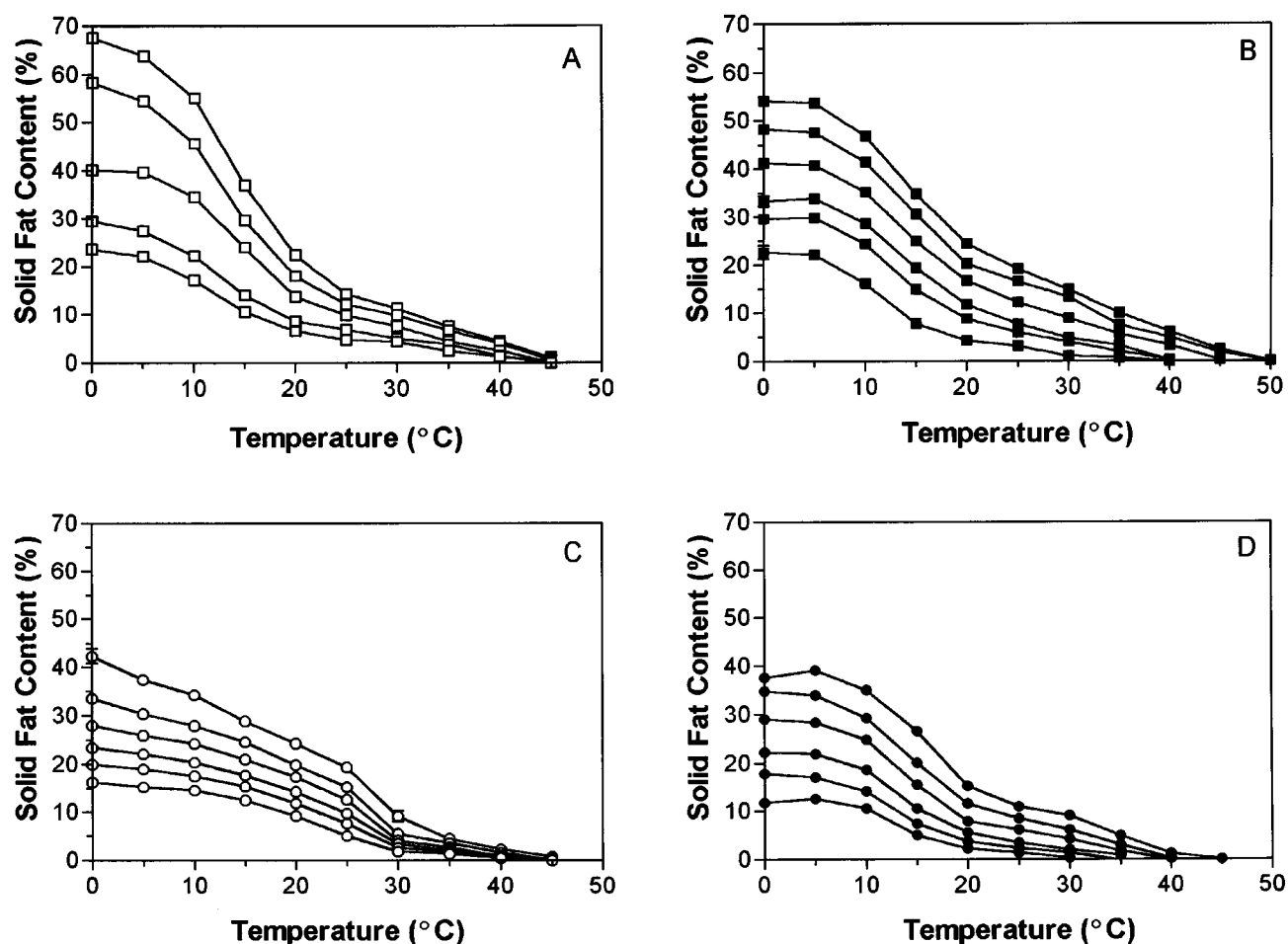


FIG. 3. Changes in the solid fat content of palm oil-soybean oil (POSBO) and lard-canola oil (LCO) blends as a function of temperature ( $^{\circ}\text{C}$ ); (A) noninteresterified (NIE) POSBO blends; (B) chemically interesterified (CIE) POSBO blends; (C) NIE LCO blends; (D) CIE LCO blends. Open symbols correspond to NIE blends while solid symbols correspond to CIE blends.

PO had an SFC of 68% at  $0^{\circ}\text{C}$ , with a  $\sim 30\%$  decrease between 10 and  $20^{\circ}\text{C}$  (Fig. 3A). Dilution of PO with SBO gradually lowered initial SFC at  $0^{\circ}\text{C}$ , which continued until the end of melt. CIE linearized PO and 90% PO blend melting profiles by lowering SFC  $\leq 15^{\circ}\text{C}$  and increasing it  $\geq 20^{\circ}\text{C}$ , analogous behavior of milk fat to CIE (Fig. 3B) (10). With 80% or less PO, SFC was lowered from  $0^{\circ}\text{C}$  to the end of melting due to CIE.

The SFC of native lard (Fig. 3C) decreased linearly from 0 to  $25^{\circ}\text{C}$ . Dilution with CO gradually led to the appearance of two distinct melting zones,  $0\text{--}10^{\circ}\text{C}$  and  $15\text{--}25^{\circ}\text{C}$ , possibly indicating increased intersolubility of the lower-melting ( $0\text{--}10^{\circ}\text{C}$ ) TAG of lard in the CO. This may be explained by the greater likelihood of finding unsaturated fatty acids in the lower-melting TAG of lard than in the higher-melting TAG. CIE of lard and LCO blends led to less linear profiles and narrowed the melting range substantially (Fig. 3D). For example, the SFC of native lard at  $25^{\circ}\text{C}$  was  $\sim 20\%$ , whereas that of CIE lard was  $\sim 10\%$ . Others have shown that the physical properties of lard can be substantially altered by CIE (16).

**TAG solution behavior.** The isosolid diagrams of NIE and CIE POSBO and LCO (Fig. 4) indicate the monotectic solu-

tion behavior, which is prevalent in mixed systems that consist of fats with widely different melting points (17). Monotectic behavior occurs when the higher-melting TAG of a fat are solubilized in the liquid TAG component (18). For example, the gradually lower than expected temperature observed for 5% SFC for the POSBO system (Fig. 4A) with increased oil content indicates greater intersolubility of PO in SBO as the proportion of oil is increased. Furthermore, the generally lower temperatures for the CIE blends (both POSBO and LCO) indicate greater solubility of the higher-melting TAG in the liquid after interesterification (Fig. 4B and 4D, respectively). The mixing behavior of tripalmitin and tristearin with triolein was discussed by Norton *et al.* (19), who also observed changes in solubility.

Our results suggest that TAG intersolubility strongly influences the mechanical properties of the fat crystal network because there is a substantial lard and PO TAG solubilization in the CO and SBO, respectively. This solubilization increases relatively as the proportion of oil is increased in the blends, as shown in previous work (20). For example, PO has an SFC of  $\sim 68\%$  at  $5^{\circ}\text{C}$ . Assuming negligible PO solubility in SBO, we would predict an SFC of  $\sim 34\%$  for a blend of 50% PO and

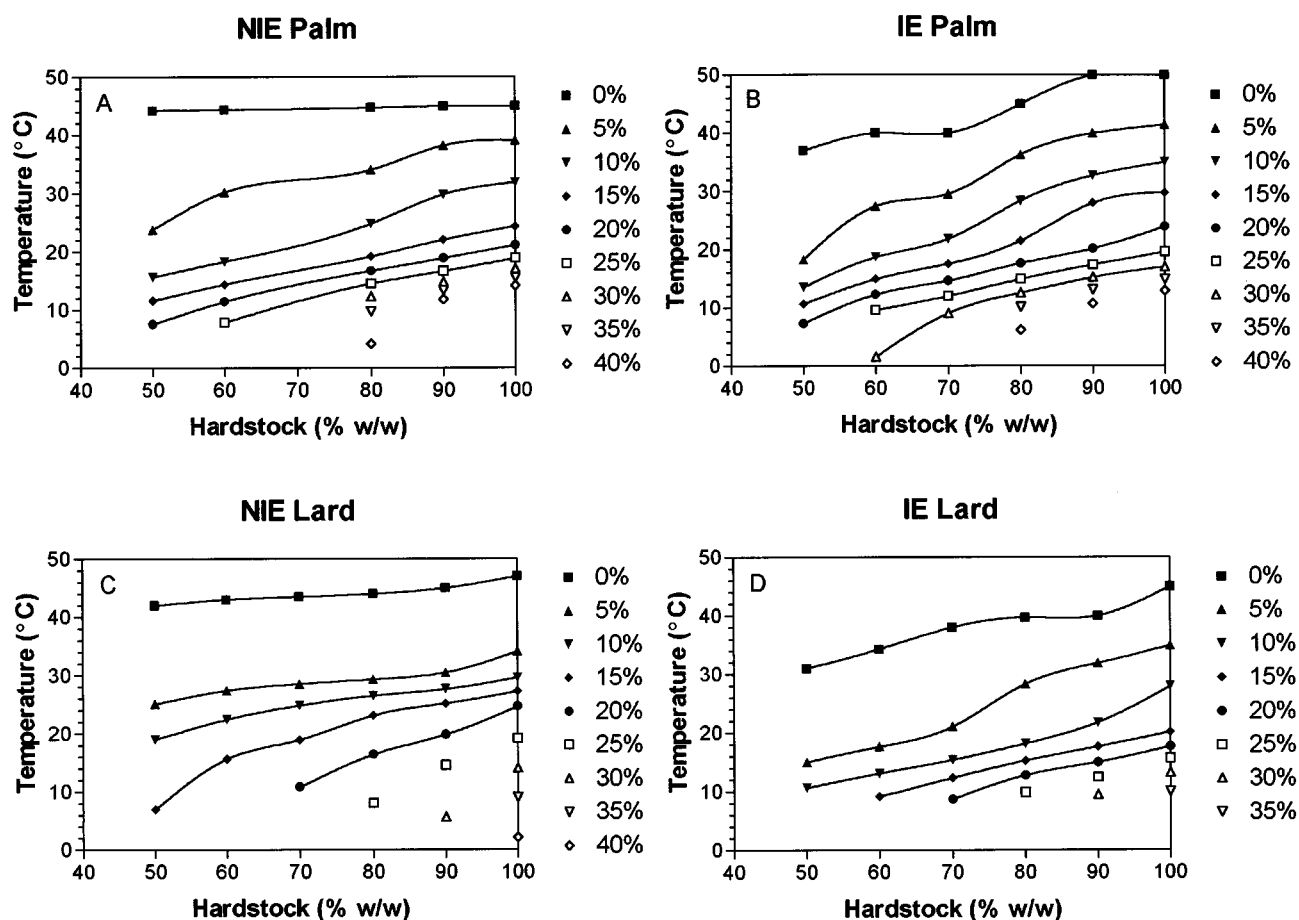


FIG. 4. Isosolid lines for POSBO and LCO blends. (A) NIE POSBO; (B) CIE POSBO; (C) NIE LCO; (D) CIE LCO. See Figure 3 for abbreviations.

50% SBO; however, the measured SFC was ~23%. This decrease in the amount of supersaturation will affect crystallization behavior. The decreased final SFC will also lead to a weaker network and impaired oil holding capacity.

**Crystallization behavior.** Crystallization consists of two steps: nucleation and crystal growth (21). Modification of a hardstock *via* blending and/or CIE can alter the kinetics of both of these processes. LCO blend peak crystallization temperatures (°C) decreased with addition of 10% (w/w) CO (Table 1). Further addition of oil did not affect peak crystallization temperature. Interesterification of the LCO blends increased crystallization peak temperatures for the LCO blends with 80% (w/w) and 90% (w/w) lard. Peak temperatures of blends with 60% (w/w) or 70% (w/w) CO decreased relative to their NIE counterparts as a result of CIE. The crystallization peak temperature of pure lard was not affected by CIE (21.4 vs. 21.2°C)—an unexpected result, given the noticeable change in TAG distribution after interesterification. PO crystallization temperatures were generally higher for the interesterified blends than for their NIE counterparts and not greatly affected by addition of SBO (Table 1). Variability was observed, though, in all these general trends.

Table 2 outlines the differences between crystallization onset and peak temperatures ( $\Delta T_{o-p}$ ) for the NIE and CIE LCO and POSBO blends. This parameter is directly proportional

to the rate of crystal growth. As shown in Table 2, the  $\Delta T_{o-p}$  is larger for the CIE blends than for the NIE blends in both systems. As postulated by Rousseau *et al.* (11), this results from the randomization of saturated fatty acids among all TAG species present, potentially leading to increased nucleation and decreased crystallization rates. In terms of crystal packing, the random rearrangement of fatty acids in the TAG

TABLE 1  
Effect of Blending and Chemical Interesterification on Crystallization Peak Temperatures (°C) of Palm Oil–Soybean Oil and Lard–Canola Oil Blends as Measured by Differential Scanning Calorimetry

Hardstock in blend (% w/w)	Lard–canola oil blends		Palm oil–soybean oil blends	
	NIE (°C)	CIE (°C)	NIE (°C)	CIE (°C)
100	21.4 <sup>a,1</sup>	21.2 <sup>a,1</sup>	18.3 <sup>a,1</sup>	21.9 <sup>b,1</sup>
90	18.0 <sup>a,2</sup>	22.6 <sup>b,1</sup>	18.8 <sup>a,1</sup>	21.0 <sup>b,1</sup>
80	18.5 <sup>a,2</sup>	21.0 <sup>b,1</sup>	21.2 <sup>a,1</sup>	19.3 <sup>b,1</sup>
70	17.1 <sup>a,1,2</sup>	15.8 <sup>a,2</sup>	18.9 <sup>a,1</sup>	20.0 <sup>b,1</sup>
60	21.6 <sup>a,3</sup>	13.5 <sup>b,3</sup>	16.0 <sup>a,1</sup>	16.5 <sup>a,2</sup>

<sup>a,b</sup>Comparisons between noninteresterified (NIE) and chemically interesterified (CIE) treatments, separately for each lard and palm oil blend. Means with the same letter are not significantly different as determined by the protected LSD test ( $P > 0.05$ ).

<sup>1,2,3</sup>Comparisons between blends within a column. Means with the same number are not significantly different as determined by the protected LSD test ( $P > 0.05$ ).

**TABLE 2**  
Difference Between Onset and Peak Crystallization Temperatures of Noninteresterified and Chemically Interesterified Palm Oil-Soybean Oil and Lard-Canola Oil Blends

Hardstock in blend (% w/w)	Lard-canola oil blends		Palm oil-soybean oil blends	
	$\Delta T_{o-p}$ NIE ( $^{\circ}\text{C}$ )	$\Delta T_{o-p}$ CIE ( $^{\circ}\text{C}$ )	$\Delta T_{o-p}$ NIE ( $^{\circ}\text{C}$ )	$\Delta T_{o-p}$ CIE ( $^{\circ}\text{C}$ )
100	1.07 <sup>a,1</sup>	2.00 <sup>a,1</sup>	0.60 <sup>a,1</sup>	1.13 <sup>b,1</sup>
90	1.03 <sup>a,1</sup>	1.50 <sup>b,1</sup>	0.93 <sup>a,1</sup>	1.67 <sup>a,1</sup>
80	1.03 <sup>a,1</sup>	1.70 <sup>a,1</sup>	0.93 <sup>a,1</sup>	1.37 <sup>b,1</sup>
70	0.57 <sup>a,1</sup>	1.80 <sup>a,1</sup>	1.23 <sup>a,1</sup>	0.93 <sup>a,1</sup>
60	0.83 <sup>a,1</sup>	1.03 <sup>a,1</sup>	1.17 <sup>a,1</sup>	1.33 <sup>a,1</sup>

<sup>a,b</sup>Comparisons between noninteresterified NIE and CIE treatments, separately for each lard and palm oil blend. Means with the same letter are not significantly different as determined by the protected LSD test ( $P > 0.05$ ). See Table 1 for abbreviations.

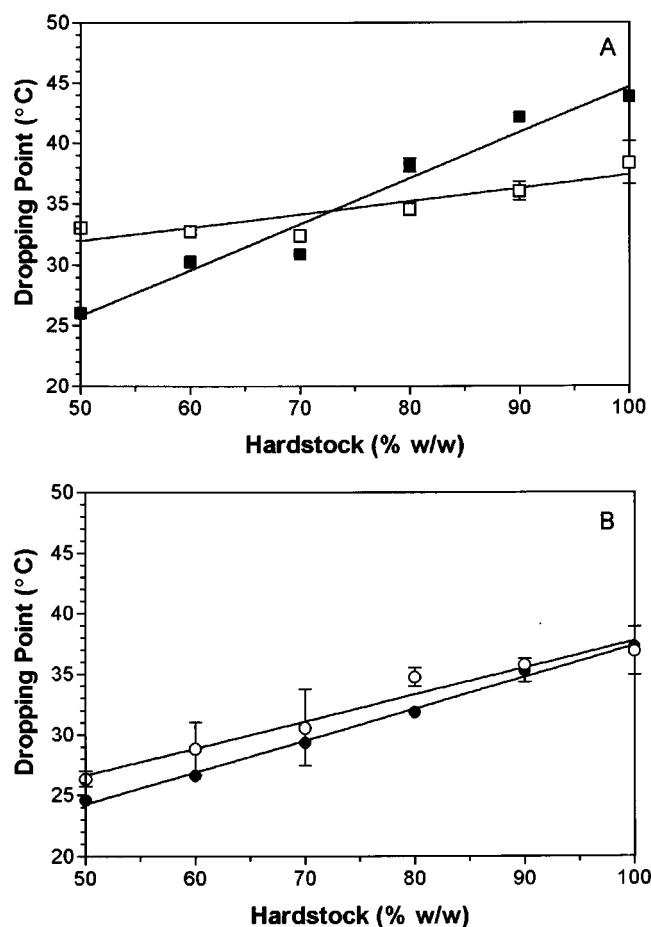
<sup>1,2</sup>Comparisons between blends within a column. Means with the same number are not significantly different as determined by the protected LSD test ( $P > 0.05$ ).

would destroy any structural complementarity among TAG, which would favor crystal growth. CIE in both PO and lard results in kinetically and/or thermodynamically less favorable crystal growth.

**Dropping point (DP).** DP measurements have traditionally been used to study melting behavior of fats. Changes in DP in the POSBO and LCO systems are shown in Figures 5A and 5B. The measured DP of PO was 38 $^{\circ}\text{C}$ , similar to other studies (22,23). CIE increased PO DP by 5.5 $^{\circ}\text{C}$ . Others have also found that CIE increased PO DP (23). CIE POSBO blends with  $\geq 80\%$  PO in the blend had higher DP and lower DP with  $\leq 70\%$  in the blend compared to NIE blends. From 100% PO to 50% PO in the blend, there was a 5–6 $^{\circ}\text{C}$  drop for NIE blends and an 18 $^{\circ}\text{C}$  drop for CIE blends.

The change in DP for PO can be correlated to changes in both SFC and TAG composition. CIE increased the content of 48-carbon species by  $\sim 5\%$ , which are likely trisaturated palmitic acid TAG. For both POSBO and LCO systems, changes in DP could be correlated to changes in SFC profiles. For example, native PO had a DP of 38 $^{\circ}\text{C}$ , which increased to 44 $^{\circ}\text{C}$  after CIE. Figures 6A and 6B show the SFC vs. temperature profiles of PO and lard, respectively. Upon CIE, the SFC of PO around its DP temperature is higher. This explains the increase in DP for palm oil upon CIE. No differences in SFC were noticed for lard upon CIE, in agreement with the absence of change in its DP. For the 50% blend of POSBO (Fig. 7A), we notice decreases in SFC upon CIE, at temperatures in the range of its DP, in agreement with decreases in its DP (Fig. 5A). The same trend was observed for the 50% LCO blend (Fig. 7B), in agreement with a decreased DP upon CIE (Fig. 5B).

DP of NIE and CIE LCO blends were similar, with differences of 0.4–1.6 $^{\circ}\text{C}$  between all NIE and CIE equivalents, indicating that CIE did not greatly affect lard DP. Luddy *et al.* (24) and Kaufmann and Grothues (25) also found that CIE of lard did not affect its melting point. Incremental dilution of lard with CO lowered DP by 10.5 $^{\circ}\text{C}$  for NIE blends and



**FIG. 5.** Changes in dropping point ( $^{\circ}\text{C}$ ) for NIE POSBO blends (A) and LSO blends (B). Error bars represent standard deviations of three replicates. Open symbols correspond to NIE blends while solid symbols correspond to CIE blends. See Figure 3 for abbreviations.

12.7 $^{\circ}\text{C}$  for CIE blends, again indicating greater LCO TAG inter-solubility for the CIE blends.

Interesterification leads to partial or total randomization of TAG present within a fat or fat blend. This randomization produces more similar TAG, which then become more inter-soluble as they are more similar. Hence, it is easier for inter-esterified fat blends to form solid solutions (mixed crystals). Solid solution formation does not readily occur between single acid and symmetrical-mixed TAG (26). Production of more similar crystals leads to lowered DP. As with other systems, the blending of a hardstock with large amounts of oil ( $\geq 30\%$  oil) and subsequent interesterification will lower the DP or melting point (10,27).

The changes in DP in the blends after CIE could not be directly related to the changes in composition. CIE of lard and LCO blends led to greater TAG compositional changes than CIE of PO and POSBO blends, but to a smaller difference in DP than for the PO blends. Furthermore, each system was influenced differently by the addition of oil. The POSBO systems underwent only a 5–6 $^{\circ}\text{C}$  drop between 100 and 50% PO in the blend, while lard blend DP diminished by 9–10 $^{\circ}\text{C}$

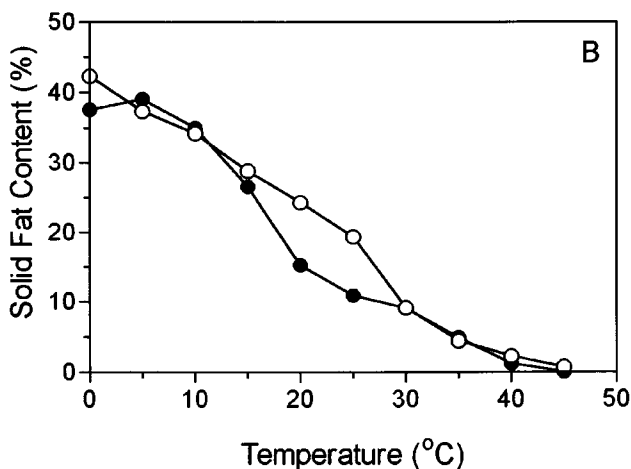
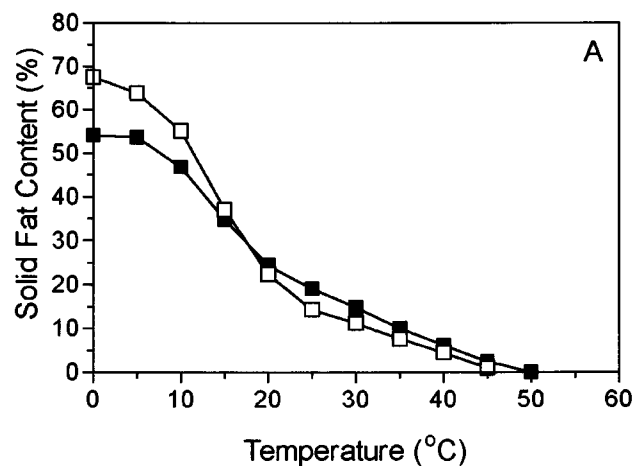


FIG. 6. Change in the SFC of palm oil (A) and lard (B) as a function of temperature ( $^{\circ}\text{C}$ ). Open symbols correspond to NIE blends while solid symbols correspond to CIE blends. See Figure 3 for abbreviations.

within the same range. From a solubility standpoint, this indicates greater compatibility of the lard with CO than of PO with SBO because the lard TAG were more soluble in the oil than the PO TAG. In a study of the physical properties of interesterified fat blends, Zeitoun *et al.* (28) noted the impact of the liquid component on the physical properties of plastic fats.

The DP of fats corresponds to an SFC of 3–5%. Studies of water-in-oil emulsions have shown that coalescence of the aqueous phase does not occur with as little as 3–4% SFC (29). Structurally, fats can form a network even at low SFC. This behavior was evident in all blends before and after interesterification. This would seem to indicate that a continuous fat crystal network is present and is responsible for much of the solid-like behavior of plastic fats. Without the presence of such a network, the DP would be expected to be much lower.

In summary, the physical properties of both the lard and PO systems were greatly influenced by blending and CIE. There is a lack of correlation between changes in DP and composition as small changes in composition led to large changes in DP. This indicates that composition cannot be used as sole predictor of physical properties such as melting be-

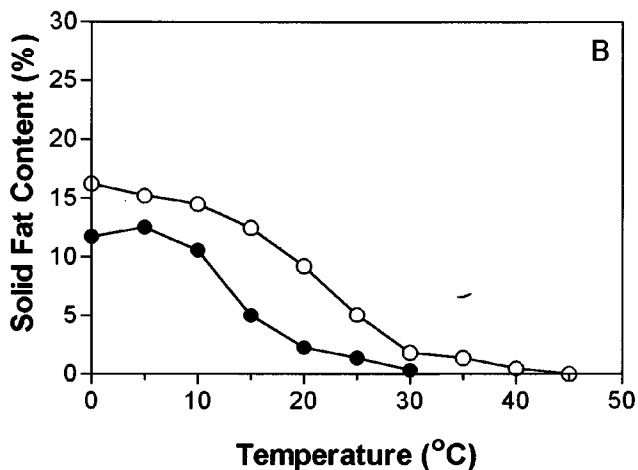
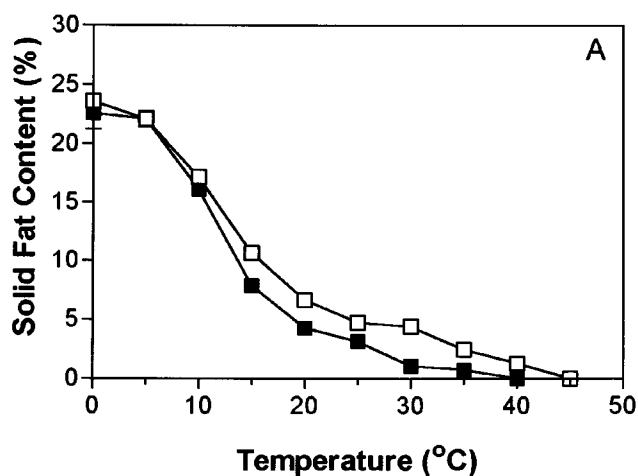


FIG. 7. Change in the SFC of 50% (w/w) POSBO (A) and 50% (w/w) LSO blends (B) as a function of temperature ( $^{\circ}\text{C}$ ). Open symbols correspond to NIE blends while solid symbols correspond to CIE blends. See Figure 3 for abbreviations.

havior. Prediction of DP and SFC of complex fats must also take the solubility effect into consideration because increased TAG intersolubility can substantially lower SFC and DP and quite possibly weaken the fat crystal network.

The effects of CIE on crystal habit, rheological properties, and the structure of the fat crystal network in PO and lard will be presented in parts 2 and 3 of this series.

#### ACKNOWLEDGMENTS

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