DSC Study on the Melting Properties of Palm Oil, Sunflower Oil, and Palm Kernel Olein Blends Before and After Chemical Interesterification

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ABSTRACT: Changes in DSC melting properties of palm oil (PO), sunflower oil (SFO), palm kernel olein (PKOo), and their blends in various ratios were studied by using a combination of blending and chemical interesterification (CIE) techniques and determining total melting (ΔH_f) and partial melting ($\Delta H_{i°C}$) enthalpies. Blending and CIE significantly modified the DSC melting properties of the PO/SFO/PKOo blends. PO and blends containing substantial amounts of PO and PKOo experienced an increase in their DSC ΔH_f and $\Delta H_{i°C}$ following CIE. The DSC ΔH_f and $\Delta H_{i°C}$ of PKOo, blends of PO/SFO at 1:1 and 1:3 ratios, and all blends of PKOo/SFO significantly decreased after CIE. The DSC ΔH_f and $\Delta H_{i°C}$ of SFO changed little following CIE. Randomization of FA distribution within and among TAG molecules of PO and PKOo led to modification in TAG composition of the PO/PKOo blends and improved miscibility between the two fats and consequently diminished the eutectic interaction that occurred between PO and PKOo.

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KEY WORDS: Chemical interesterification, DSC melting and partial melting enthalpies, palm kernel olein, palm oil, sunflower oil.

Most native fats and oils have limited applications in their unmodified forms, imposed by their TAG and FA compositions. To widen their use, they may be modified by blending, fractionation, interesterification (IE), hydrogenation, or combinations of these processes (1). Changing the natural physical characteristics of a fat or oil offers greater functionality for a large number of product formulations (2,3).

Blending is the simplest modification. As requirements for fat products become more sophisticated, however, there are correspondingly fewer ways that appropriate specifications can be met by simply blending natural fats and oils. Other modification techniques need to be used for one or more of the blend components to meet product specifications at the lowest cost (3). Hydrogenation of polyunsaturated vegetable oils is currently less appealing because of evidence that *trans* FA have adverse nutritional effects (4,5). IE has received much attention in the edible oil industry as an alternative method to improve the physical properties of fats and oils.

IE involves interchanging FA within and between TAG molecules that make up fats and oils and leads to an increased number of TAG types. Thus, important physicochemical properties, such as melting and crystallization behaviors of the fats and oils, are modified. The precise effect of IE on the properties of fat and oil depends very much on the type of starting material that is subjected to IE. For instance, the m.p. and solid fat content of a high-melting fat would be reduced when it is interesterified (IEed) with a liquid oil (2). Two types of IE are currently in commercial use, i.e., chemical (CIE) and enzymatic (EIE). CIE and random EIE using nonspecific lipases lead to a random distribution of FA on the TAG (6). In contrast, in using a 1,3-specific lipase, only the FA in the 1,3-positions are shifted; the 2-position is unchanged. Preservation of the 2-position means that a more natural fat is produced and results in the production of a much smaller absolute number of TAG compared with CIE and random EIE. Generally, EIE has certain advantages over CIE, such as milder reaction conditions and regiospecificity (7), and is currently a cost-effective alternative to CIE (8).

Palm oil (PO), an important edible oil source for the food industry, tends to crystallize as β′ and therefore is an attractive option for the production of yellow fat spreads. For the production of such products, however, PO is normally combined with other fats and/or oils because PO alone does not result in products that melt quickly on the palate (9,10). Hence, PO may be blended and/or IEed with a lauric fat that contains short- and medium-chain FA and/or a liquid vegetable oil to give blends with improved melting properties. Lauric fats are often used as one component of a blend to provide a broader spectrum of chain lengths, to provide a concentrated source of saturates, or to achieve both goals. A liquid vegetable oil is always used to provide a source of unsaturated FA (3).

The aim of the present work was to investigate the effects of blending and CIE on the melting behavior of PO, sunflower oil (SFO), palm kernel olein (PKOo), and blends thereof in various ratios. The melting behaviors of the oils/fats and their blends were studied by using their DSC melting thermograms. The DSC melting thermogram gives valuable information on the melting profile of fats, e.g., on how they melt in the mouth during mastication. It can also be used to monitor the physical

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interaction of fats in blends from the changes in thermal characteristics, such as the total melting (ΔH_f) and partial melting ($\Delta H_{i°C}$) enthalpy. DSC ΔH_f of an oil or fat is the total energy required to bring the substance from the solid state to a complete melt. DSC $\Delta H_{i°C}$ of an oil or fat is the energy required to bring the substance from the solid state at the said temperature to a complete melt (11). The interactions of these lipids and their compatibility due to blending and CIE are also discussed

EXPERIMENTAL PROCEDURES

CIE Blends preparation. Blends of PO, SFO and PKOo were prepared in the following mass (w/w) ratios: A[1:0:0], B[3:1:0], C[1:1:0], D[1:3:0], E[0:1:0], F[0:3:1], G[0:1:1], H[0:1:3], I[0:0:1], J[1:0:3], K[1:0:1], L[3:0:1], M[4:1:1], N[1:4:1], P[1:1:4] and Q[1:1:1]. Fat blends (250 g) were dried for 30 min at 110°C under a steady stream of nitrogen, and 0.2% sodium metal was added as catalyst. After 60 min of stirring at a constant speed of 2000 rpm, the mixture was cooled to 60–70°C and then 20% citric acid solution was added to deactivate the catalyst. The mixture was then transferred to a separatory funnel with a bottom opening for washing. Excess hot water was added, the mixture was stirred mechanically for 15–20 min and was then allowed to settle for 10 min to separate the oil and the water phase before discarding the water phase. The washing process was repeated several times to ensure that the sample was completely free from citric acid, catalyst residue, and the soaps resulting from the reaction between sodium ion and any free FA present or produced during the process. Finally, the IEed fat blend was dried under vacuum at 110°C for approximately 30 min.

FA composition (FAC). FAC was determined as FAME. FAME were prepared by dissolving 50 mg of fat blend in 950 µL hexane (Merck, Darmstadt, Germany), in a 2-mL screw-capped vial, to which was added 50 µL of 1 M sodium methoxide (Merck). The mixture was then mixed vigorously for 1 min or until it became cloudy. Distilled water (1 mL) was added, and the mixture was thoroughly mixed. It was then allowed to settle and separate into two distinct layers. The upper clear supernatant, i.e., the FAME, was decanted, and a small amount of anhydrous sodium sulfate (Merck) was added to remove traces of moisture. FAME (1 µL) was injected into a gas chromatograph fitted with a polar SP 2340 fused-silica capillary column (60 m length × 0.25 mm i.d. × 0.2 µm film thickness) (Supelco, Bellefonte, PA). An FID was used to detect the FA. The detector and injector temperatures were set at 240°C. The carrier gas was helium at 0.8–1.0 mL/min. The column temperature was isothermal at 185°C for PO and SFO. The column temperature for PKOo was programmed (nonlinear) at 120–185°C at 3°C/min.

DSC melting behavior. The DSC melting properties were studied using a Pyris Series DSC 7 Differential Scanning Calorimeter. Calibration was performed with *n*-decane (m.p. –29.66°C, ΔH_f 202.09 J/g) and indium (m.p. 156.60°C, ΔH_f 28.45 J/g). Approximately 7 to 8 mg of a precisely weighed (±0.005 mg) fat sample was placed in the sealed DSC pan and transferred to the DSC head. The samples were heated at 70°C for 15 min to destroy crystal memory, cooled at 0°C for 90 min and at −50°C for 5 min. DSC melting curves were then recorded at a heating rate of 10°C/min from −50 to 60°C. An empty covered sample pan was used as the reference. The $\Delta H_{i°C}$ was calculated by means of the DSC Data Analysis software.

Statistical analysis. Analyses of general linear models and response surfaces were performed using Design-Expert Version 6.0.4 (Stat-Ease Inc., Minneapolis, MN). R^2 values, which indicate model fits for each of the constructed ternary diagrams, were determined and found to be greater than 0.97.

RESULTS AND DISCUSSION

The DSC ΔH_f and $\Delta H_{i°C}$ values for the initial and chemically interesterified (CIEed) PO (coded A), SFO (coded E), PKOo (coded I), and their blends in various ratios (coded C-D, F-H, and J-Q) are presented in Table 1.

Individual oils. PKOo, a lauric-rich fat, had the highest ΔH_f (106.7 J/g), followed by PO (78.8 J/g) and SFO (20.6 J/g). Based on the DSC thermal characteristics, it was observed that though PKOo had higher ΔH_f and $\Delta H_{i°C}$ at the lower temperature range (5–20°C), it completely melted at 25–30°C, lower than the complete melting temperature for PO. Complete melting temperature for PO was 35–40°C, much higher than PKOo, owing to the presence of a high proportion of long-chain saturated FA, mainly palmitic and to a lesser extent stearic acid. The sharp melting profile of PKOo is related to the high content of short- and medium-chain saturated caprylic, capric, lauric, and myristic FA. The ΔH_f of SFO was very low as very little energy was needed to melt the already liquid oil, attributed to the presence of a high content of mono- and polyunsaturated FA. The higher the degree of unsaturation of a FA, the lower the m.p. is of a TAG (12). The lower the m.p. of a TAG, the softer the fat containing such TAG is, as the softness of a fat is related to its TAG composition, which in turn is related to its corresponding FAC. The FAC of PO, SFO, and PKOo is shown in Table 2.

PO experienced a significant increase in ΔH_f and $\Delta H_{i°C}$, and consequently an increase in hardness, following CIE. This was due to the increase in TAG composed of three saturated FA (S_3), i.e., tripalmitin (PPP) and dipalmitostearin (PPS), where P = palmitic and S = stearic acids, and a simultaneous decrease in some disaturated-monounsaturated (S_2U) (PLP, POP, and POS) and diunsaturated-monosaturated (U_2S) (POO and SOO) TAG, where L = linoleic and O = oleic acids, as reported by Noor Lida *et al.* (13). This finding is also consistent with the review of Allen (3), wherein most IEed oils have higher m.p. after IE, owing to the formation of S_3 TAG. The melting characteristics of SFO were hardly altered after CIE, presumably due to the fact that SFO is very rich in unsaturated long-chain FA. Thus, randomization did not result in much change in the TAG composition of SFO. CIE slightly reduced ΔH_f and $\Delta H_{i°C}$, of PKOo. This could have been due to the decrease in some of the S_3 TAG, e.g., CLaLa, CaLaLa/CLaM, and LaLaLa (where C = capric, La = lauric, Ca = caprylic, and M = myristic acids) and a simultaneous increase in TAG species with intermediate

TABLE 1
DSC Total and Partial Melting Enthalpy of Palm Oil (PO), Sunflower Oil (SFO), and Palm Kernel Olein (PKOo) and Their Blends in Various Ratios Before (DB) and After (CIE) Chemical Interesterification

Code	PO/SFO/PKOo ratios		Partial melting enthalpy ($\Delta H_{i°C}$) (J/g) 5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	Total melting enthalpy (ΔH_f) (J/g)
A	1:0:0	DB	53.4	41.9	37.0	30.7	25.3	18.4	12.7	3.0	84.8
		CIE	64.1	56.7	50.7	46.8	39.1	29.1	18.9	9.3	93.4
B	3:1:0	DB	36.0	27.8	17.8	16.0	14.4	10.0	4.4	0.1	77.2
		CIE	39.0	29.6	20.8	17.1	15.5	10.8	5.1	0.3	76.8
C	1:1:0	DB	19.5	14.4	11.1	7.7	6.0	3.6	0.8	—	71.0
		CIE	22.9	16.2	10.7	7.1	4.6	2.3	0.2	—	68.3
D	1:3:0	DB	9.5	6.8	3.8	3.0	1.8	0.3	—		63.6
		CIE	10.4	6.2	3.0	1.4	0.3	—			56.0
E	0:1:0	DB	0.7	0.3	—						20.6
		CIE	0.8	0.2	—						33.1
F	0:3:1	DB	18.7	10.1	5.9	0.9	—				67.3
		CIE	7.6	2.7	0.3	—					59.9
G	0:1:1	DB	42.7	32.9	20.2	5.5	—				78.3
		CIE	27.0	17.4	6.5	0.3	—				62.2
H	0:1:3	DB	62.3	54.6	43.1	26.1	2.0	—			96.6
		CIE	57.8	47.2	32.7	13.9	0.3	—			86.8
I	0:0:1	DB	88.5	79.7	67.1	49.6	21.6	0.1	—		106.7
		CIE	81.2	72.5	59.3	42.6	17.3	—			102.4
J	1:0:3	DB	75.5	65.1	51.9	34.5	6.2	0.7	0.1	—	101.6
		CIE	79.8	72.0	59.1	44.1	23.8	2.3	—		107.1
K	1:0:1	DB	62.6	51.9	36.7	20.4	7.1	2.9	0.8	0.1	90.5
		CIE	61.9	55.1	45.0	33.6	24.2	10.1	0.2	—	97.7
L	3:0:1	DB	51.2	39.9	24.0	17.5	13.5	8.8	4.1	0.1	81.3
		CIE	68.9	57.5	47.1	39.8	31.8	20.2	8.3	0.2	98.5
M	4:1:1	DB	40.5	29.8	18.9	15.0	11.4	7.1	2.8	0.1	81.9
		CIE	44.4	37.8	28.9	24.1	17.1	9.5	2.4	—	82.0
N	1:4:1	DB	14.7	9.3	4.2	2.1	1.3	0.4	0.1	—	67.8
		CIE	11.9	6.6	3.0	0.9	—				53.9
P	1:1:4	DB	68.6	57.5	42.3	22.1	1.9	0.1	—		89.0
		CIE	54.4	46.4	35.9	23.8	6.6	0.1	—		88.9
Q	1:1:1	DB	35.4	27.0	15.3	6.2	3.6	1.8	0.4	—	74.5
		CIE	35.8	25.9	17.2	9.9	4.5	0.2			73.6

degrees of unsaturation, e.g., LaLaO and LaOM. The CLaLa, CaLaLa/CLaM, and LaLaLa were reduced from 8.4, 11.8, and 22.7% before CIE to 5.5, 6.6, and 18.6%, respectively, after CIE. On the other hand, the LaLaO and LaOM were increased from 6.4 and 5.0% before CIE to 13.6 and 7.6% after CIE (13). The effect of CIE on $\Delta H_{i°C}$ of PO, PKOo, and SFO at various temperatures is shown in Figure 1.

Binary blends of PO/PKOo, PO/SFO, and PKOo/SFO. The ΔH_f and $\Delta H_{i°C}$ of PO and PKOo gradually and proportionally decreased with an increase in the proportion of SFO in the PO/SFO (coded B, C, and D) and PKOo/SFO (coded F, G and H) blends. The softening effect was due to the liquid triunsaturated (U_3) TAG in SFO that diluted the amounts of solid S_2U and S_3 TAG in PO and PKOo. The blends of PO/SFO and PKOo/SFO had ΔH_f and $\Delta H_{i°C}$ representing linear combinations of the two components, showing that PO and PKOo were completely miscible with SFO. On the other hand, the melting behavior of PO/PKOo blends (coded J, K, and L) did not represent linear combinations of the PO and PKOo. The $\Delta H_{i°C}$ of the blends were lower than both PO and PKOo.

TABLE 2
FA Composition of PO, SFO, and PKOo

Oils	FA[a] composition (wt%) $C_{8:0}$	$C_{10:0}$	$C_{12:0}$	$C_{14:0}$	$C_{16:0}$	$C_{18:0}$	$C_{18:1}$	$C_{18:2}$	Others
PO	—	—	0.2	1.0	42.9	4.4	40.8	10.2	0.5
SFO	—	—	—	0.1	6.3	3.7	24.3	65.1	0.5
PKOo	4.7	3.8	44.5	13.7	8.4	2.8	18.1	2.9	1.1

[a] $C_{8:0}$, caprylic acid; $C_{10:0}$, capric acid; $C_{12:0}$, lauric acid; $C_{14:0}$, myristic acid; $C_{16:0}$, palmitic acid; $C_{18:0}$, stearic acid; $C_{18:1}$, oleic acid; $C_{18:2}$, linoleic acid. "Others" includes caproic ($C_{6:0}$) and/or linolenic ($C_{18:3}$) and/or aracidic ($C_{20:0}$) acids. For other abbreviations see Table 1.

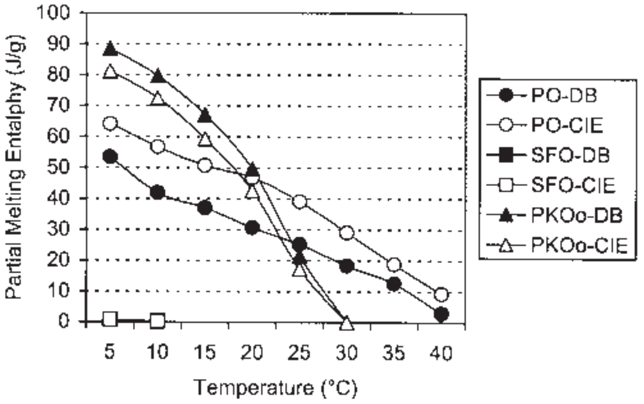

FIG. 1. Partial melting enthalpy of palm oil (PO), palm kernel olein (PKOo), and sunflower oil (SFO) before (DB) and after (CIE) chemical interesterification.

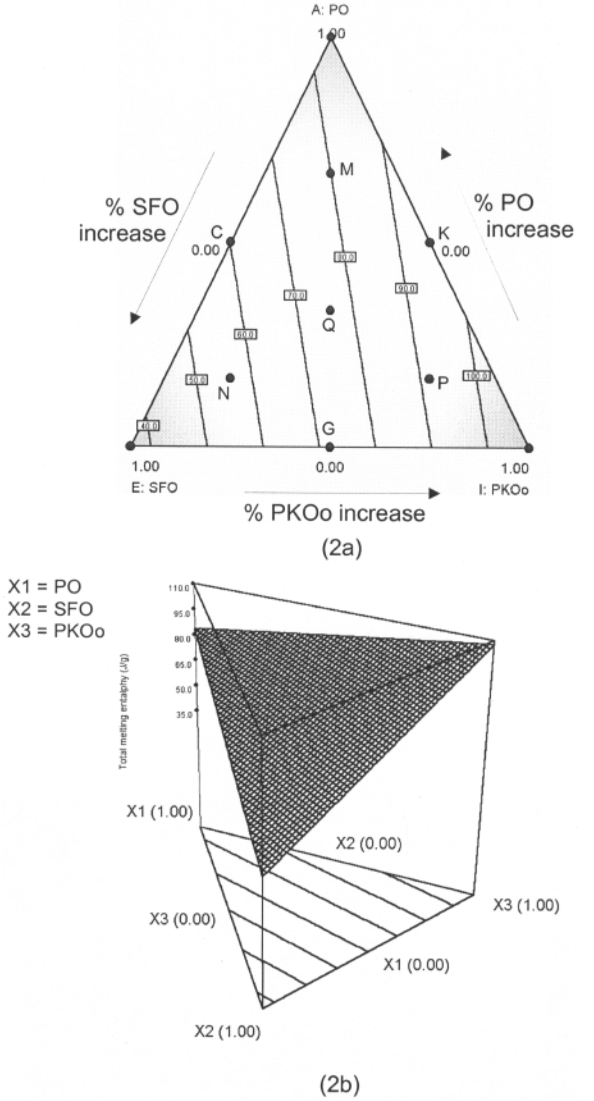

FIG. 2. (a) Contour and (b) 3-D surface diagram of total melting enthalpy of the ternary blends of PO, SFO, and PKOo. For abbreviations see Figure 1.

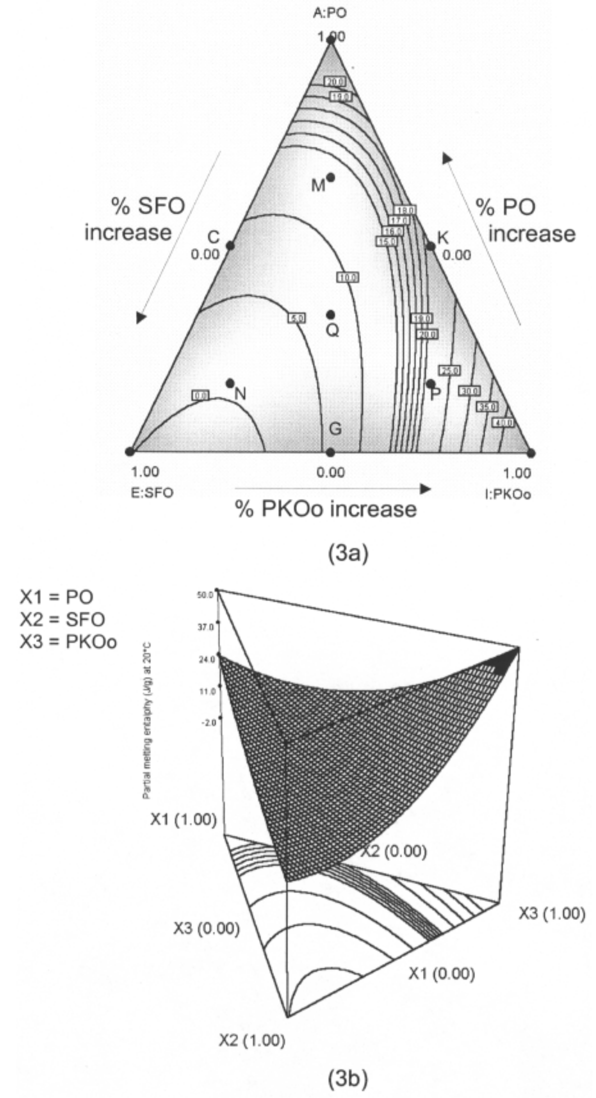

FIG. 3. (a) Contour and (b) 3-D surface diagram of partial melting enthalpy at 20°C of the ternary blends of PO, SFO, and PKOo. For abbreviations see Figure 1.

According to Dieffenbacher (14), physical characteristics of components of a fat blend that do not represent linear combinations of their components indicate the presence of interaction between the components. In this case, interaction that occurred between PO and PKOo was eutectic. Eutectic interaction is often observed in fat mixtures and is an indicator of the incompatibility between the fats. This interaction tends to occur when the fats differ in molecular volume, shape, or polymorph. A mixture with eutectic effect is softer (and consequently has lower ΔH_f and/or $\Delta H_{i°C}$) than either of the two pure fats, suggesting incompatibility between the two fats (15,16). Eutectic effects are usually undesirable, but in margarines and shortenings the effect can be beneficial (17). The eutectic interaction between PO and PKOo could not be observed from the ΔH_f of the PO/PKOo blends (Fig. 2), but was clearly evident in the $\Delta H_{i°C}$ data. Based on the $\Delta H_{i°C}$, the eutectic between PO and

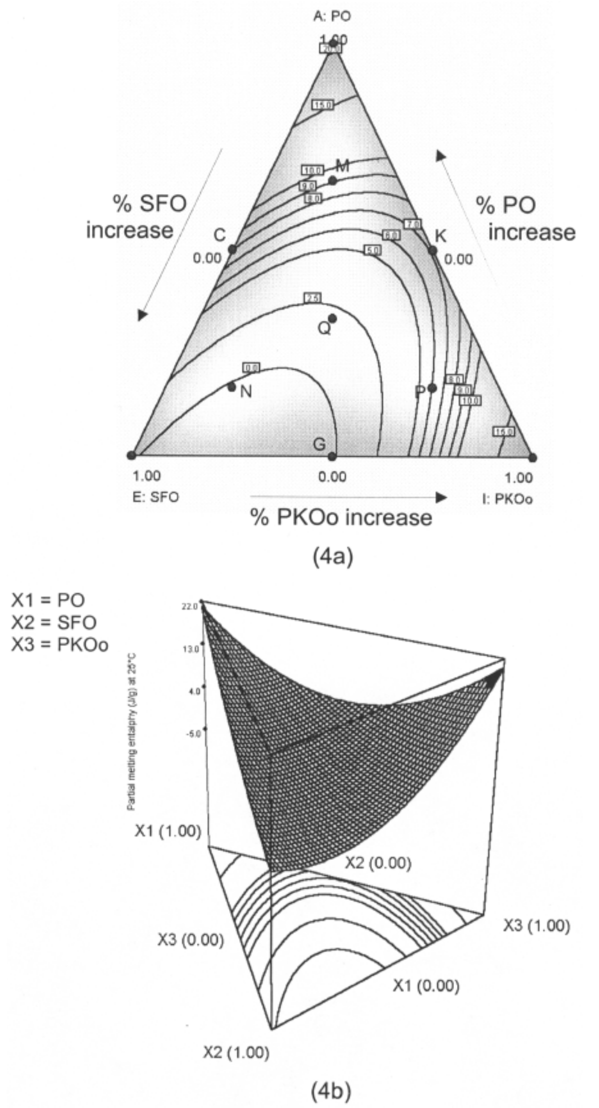

FIG. 4. (a) Contour and (b) 3-D surface diagram of partial melting enthalpy at 25°C of the ternary blends of PO, SFO, and PKOo.

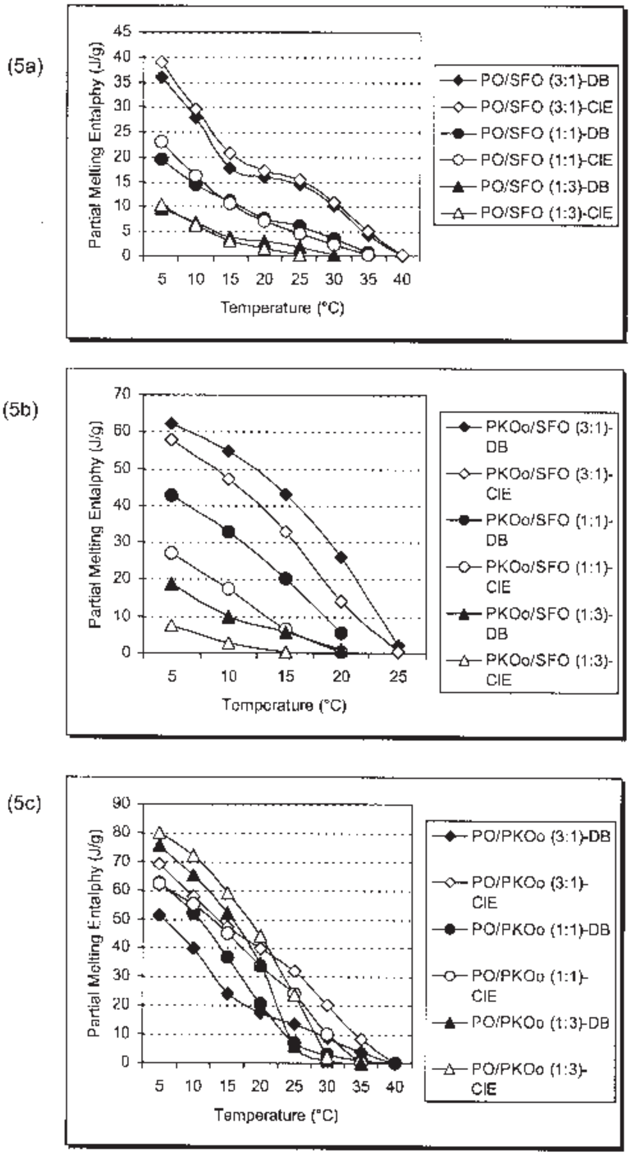

FIG. 5. Partial melting enthalpy profile of the binary blends of PO and SFO, PKOo and SFO, and PO and PKOo in various ratios before (DB) and after (CIE) chemical interesterification. For abbreviations see Figure 1.

PKOo was especially evident at 20–25°C (Figs. 3, 4). The eutectic ratio (i.e., the blend ratio with the maximum eutectic effect and, in this case, the blend that reaches its minimum $\Delta H_{i°\mathrm{C}}$), varies depending on the measurement temperature. For example, $\Delta H_{20°\mathrm{C}}$ and $\Delta H_{25°\mathrm{C}}$, i.e., the partial energy required to bring the blends from 20 and 25°C, respectively, to a complete melt showed that the eutectic ratio for the PO/PKOo blends was 3:1 at 20°C and 1:3 at 25°C. Eutectic interaction in the binary system of PO and PKOo was also described by Timms (18).

The ΔH_f and $\Delta H_{i°\mathrm{C}}$ of the binary blends of PO/SFO, PKOo/SFO, and PO/PKOo were altered following CIE, as shown in Figures 5a–c, respectively. CIEed PO/SFO at 3:1 ratio (coded B) experienced a slight increase in the $\Delta H_{i°\mathrm{C}}$ particularly at the lower temperature range. The increment in $\Delta H_{i°\mathrm{C}}$ experienced by the blend presumably was due to the trivial increase in its very high melting S_3 TAG, i.e., PPS, which increased from 0.8% before CIE to 1.3% after CIE. The m.p. of PPS is 68°C (16). However, as the proportion of SFO increases, the PO/SFO blends start to soften and consequently, the $\Delta H_{i°\mathrm{C}}$ decreases following CIE, particularly at the higher temperature range (15–35°C). This trend is also reflected in the ΔH_f of the blends and is in agreement with findings reported previously (1,19,20). The decreased ΔH_f and $\Delta H_{i°\mathrm{C}}$ values of the CIEed PO/SFO blends coded C and D, containing 50 and 75% SFO, respectively, could have been attributed mainly to the decrease in the amounts of the high-melting S_3 and S_2U TAG, mainly PPP (S_3) and POP (S_2U), concomitant with the formation of more U_2S TAG such as PLL and PLO.

Binary blends of PKOo/SFO experienced a more prominent change in their ΔH_f and $\Delta H_{i°\mathrm{C}}$ values following CIE, compared with the PO/SFO blends. The CIEed PKOo/SFO blends tended to have lower ΔH_f and $\Delta H_{i°\mathrm{C}}$ values than the non-CIEed blends.

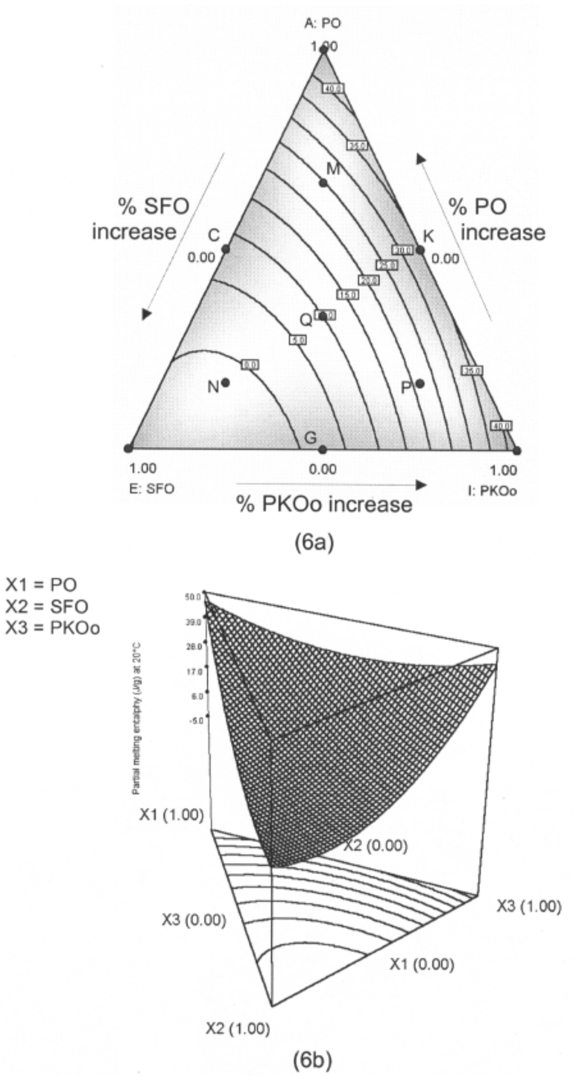

FIG. 6. (a) Contour and (b) 3-D surface diagram of partial melting enthalpy at 20°C of the chemically interesterified ternary blends of PO, SFO, and PKOo. For abbreviations see Figure 1.

Apparent changes in $\Delta H_{i°C}$ occurred at all measured temperatures. The reduced ΔH_f and $\Delta H_{i°C}$ values of the PKOo/SFO blends following CIE were attributed to the decreased proportion of the S_3 medium-chain TAG, such as LaLaLa, CaLaLa, CLaLa, and LaLaM, simultaneously with the formation of several species of low-melting TAG. The formation of the low-melting TAG in the CIEed PO/SFO and PKOo/SFO blends was due to the replacement of saturated FA in the TAG of PO and PKOo with the unsaturated FA of SFO TAG, which were mainly U_2S and U_3, as reported in Noor Lida *et al.* (13).

Binary blends of PO/PKOo also experienced a prominent change in ΔH_f and $\Delta H_{i°C}$ following CIE. The effect of CIE on melting profiles of the PO/PKOo blends was more pronounced than those of PO/SFO blends. Although the contents of high-melting S_3 and S_2U TAG, such as LaLaLa, PPP, and POP, were hardly altered following CIE, as reported by Noor Lida *et al.* (13), the binary blends of PO/PKOo experienced noteworthy increments in their ΔH_f and $\Delta H_{i°C}$. The most plausible explanation for this is that eutectic interaction between PO and PKOo in the binary blends of PO/PKOo makes the blends much softer (and less energy is needed to melt the fats) than it should be. The eutectic interaction was, however, eliminated after CIE, demonstrating a greater miscibility between the two fats, as shown by the iso-solid diagram in Figures 6 and 7. These findings are consistent with those reported by Timms (16).

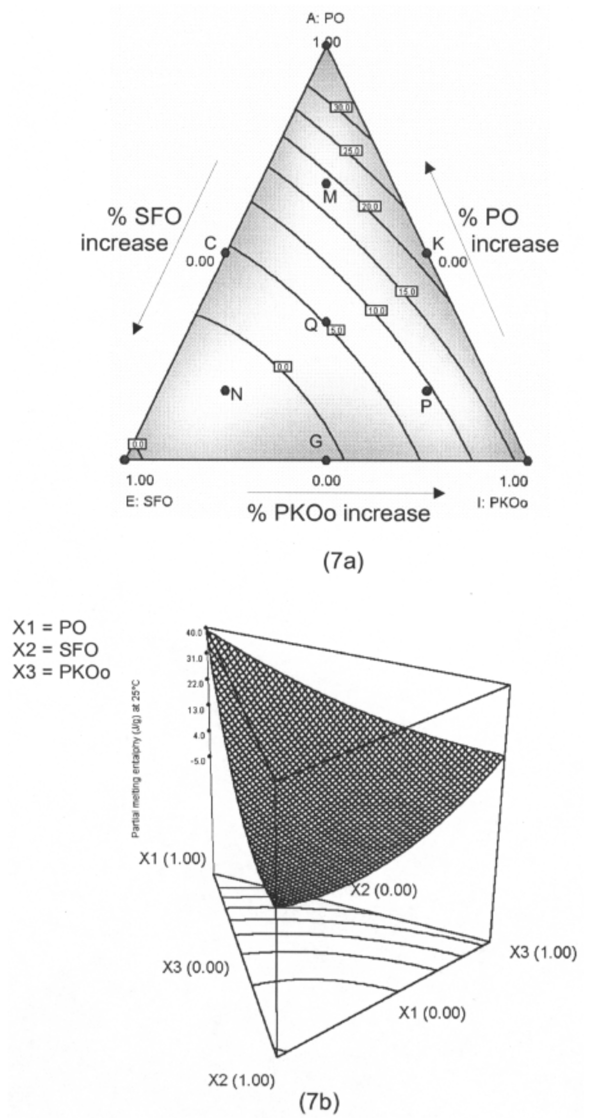

FIG. 7. (a) Contour and (b) 3-D surface diagram of partial melting enthalpy at 25°C of the chemically interesterified ternary blends of PO, SFO, and PKOo. For abbreviations see Figure 1.

Ternary blends of PO/PKOo/SFO. The melting behavior of the ternary PO/SFO/PKOo blends varied, depending on the amount of PO, SFO, and PKOo in the blends. Blends with high proportions of PKOo (66.7%, coded P) had the highest ΔH_f and $\Delta H_{i°C}$ at the low-temperature range. This result was anticipated since PKOo had high solids content at low temperature owing to the nature of its FAC. Similar to PKOo, blend P also had a much lower complete melting temperature (about 25°C) compared with the blend containing a high proportion of PO

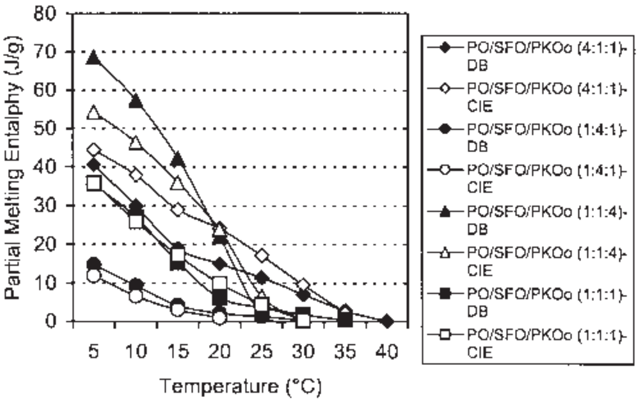

FIG. 8. Partial melting enthalpy profile of the ternary blends of PO and SFO, PKOo and SFO, and PO and PKOo in various ratios before (DB) and after (CIE) chemical interesterification. For abbreviations see Figure 1.

(66.7%, coded M). It appeared that the ternary blend coded M had the highest complete melting temperature (greater than 35°C) due to the presence of high-melting TAG in PO, e.g., PPP and POP. The ternary blend high in SFO (66.7%, coded N) was the softest blend. It had the lowest ΔH_f and $\Delta H_{i°\mathrm{C}}$ in the blend, due to the high content of PUFA contributed by SFO.

The ΔH_f and $\Delta H_{i°\mathrm{C}}$ values of CIEed ternary blends of PO/SFO/PKOo were also different from those of the starting blends (Fig. 8). CIEed ternary blend coded M appeared to have higher ΔH_f and $\Delta H_{i°\mathrm{C}}$ values (especially at 10–25°C) compared with its starting blend. This was probably due to the increase in S_3 TAG, i.e., PPP and PPS. Ternary blend coded P had lower ΔH_f and $\Delta H_{i°\mathrm{C}}$ values at the low-temperature range (5–15°C) and slightly higher values at the high-temperature range than its corresponding starting blend. The ΔH_f and $\Delta H_{\mathrm{i}°\mathrm{C}}$ of ternary blends coded N and Q (which contain equal proportion, i.e., 33.3% of PO, SFO, and PKOo) hardly changed following CIE.

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