Composition and Thermal Profile of Crude Palm Oil and Its Products

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ABSTRACT: Gas–liquid chromatography and high-performance liquid chromatography (HPLC) were used to determine fatty acids and triglyceride (TG) compositions of crude palm oil (CPO), refined, bleached, and deodorized (RBD) palm oil, RBD palm olein, and RBD palm stearin, while their thermal profiles were analyzed by differential scanning calorimeter (DSC). The HPLC chromatograms showed that the TG composition of CPO and RBD palm oil were quite similar. The results showed that CPO, RBD palm oil, RBD olein, and superolein consist mainly of monosaturated and disaturated TG while RBD palm stearin consists mainly of disaturated and trisaturated TG. In DSC cooling thermograms the peaks of triunsaturated, monosaturated and disaturated TG were found at the range of –48.62 to –60.36, –25.89 to –29.19, and –11.22 to –1.69°C, respectively, while trisaturated TG were found between 13.72 and 27.64°C. The heating thermograms of CPO indicated the presence of polymorphs β_2', α, β_1', and β_1. The peak of CPO was found at 4.78°C. However, after refining, the peak shifted to 6.25°C and became smaller but more apparent as indicated by RBD palm oil thermograms. The heating and cooling thermograms of the RBD palm stearin were characterized by a sharp, high-melting point (high-T) peak temperature and a short and wide low-melting point (low-T) peak temperature, indicating the presence of occluded olein. However, for RBD palm olein, there was only an exothermic low-T peak temperature. The DSC thermograms expressed the thermal behavior of various palm oil and its products quite well, and the profiles can be used as guidelines for fractionation of CPO or RBD palm oil.

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KEY WORDS: Crude palm oil, crystallization, DSC thermogram, fractionation, refined bleached deodorized palm oil, refined bleached deodorized palm olein, refined bleached deodorized palm stearin.

Crude palm oil (CPO) is derived from the mesocarp of the oil palm fruit (*Elaeis guineensis*). The CPO has to undergo extensive processing before it reaches the consumer. One of the processes is adsorption bleaching to produce refined, bleached, and deodorized palm oil (RBD palm oil). Fractionation is a process which separates liquid olein or superolein, used mainly for cooking oils, from solid stearin, used mainly in shortening and margarines. The CPO consists of 50% saturated and 50% unsaturated fatty acids, and the major triglycerides (TG), according to equivalent carbon number (ECN), were C_{50} (42.58%) and C_{52} (40.46%). The major TG of the olein fraction are C_{50} (42.04%) and C_{52} (45.66%) with an iodine value (IV) of about 56. The stearin fraction consists of C_{48} (22.3%), C_{50} (40%), and C_{52} (29%) TG with IV of about 44 (1). TG of palm oil consist of trisaturated (10.2%), disaturated (48%), monosaturated (34.6%), and triunsaturated (6.8%) (2).

Fractionation is accomplished by exploiting the different crystallization temperatures of the TG composing the oil. If a slow decrease of temperature is applied, then the TG molecules that have a small difference in crystallization temperatures will be crystallized at different times. This will result in a fine crystal size, and a high pressure will be needed to filter the oil. However, if a rapid temperature decrease occurs, molecules within a wide range of crystallization temperatures will be crystallized at about the same time. These crystals will agglomerate and stick to one another to form a bigger granular crystal. As a result of fast processing the olein might be occluded inside the crystal granule. Consequently, the olein yield will decrease. Therefore, control of physical properties of the oil is needed to optimize the process.

Differential scanning calorimetry (DSC) can be used to characterize the thermal behavior of edible oils. DSC studies on palm oil and palm kernel oil have been reported by several workers (3–10). Busfield and Proshogo (3,4) studied heating thermograms of palm stearin and its hydrogenation products using DSC. They showed that the heating rates and tempering time affected the profile of the thermograms and the correlation between the profile of the thermograms and crystal forms that occurred. DSC was used to support X-ray diffraction studies of oil polymorphism without reference to fractionation. Che Man and Swe (9) used DSC to study the cause of poor crystallization of a failed-batch palm oil. They found that the cooling thermogram of failed-batch palm oil differed from the one that produced good crystallization. This was due to a rapid and sudden surge of heat demand, as depicted by a rather steep slope at the $\alpha \rightarrow \beta$ phase transition.

The purpose of this work was to study the thermal behavior of palm oil and its products, and its association with the oil composition. The thermal profile and composition of CPO and its products were observed so as to be used as a guide in the fractionation process.

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MATERIALS AND METHODS

Samples. Samples of CPO, RBD palm oil, RBD palm olein, superolein, and RBD palm stearin with IV of 51.5, 51.5, 56.5, 60.0, and 30.5, respectively, were obtained from a local refinery. The IV was determined according to the AOCS method (11). All chemicals used were of analytical or high-performance liquid chromatography (HPLC) grade. All triglycerides were purchased from Sigma Chemical Co. (St. Louis, MO).

Analytical determinations. Fatty acid composition of palm oil and its products was determined through direct methylation with sodium methoxide and methanol (12). The fatty acid methyl esters obtained were then diluted with isooctane and were injected into a gas–liquid chromatograph (GLC). The methyl esters were analyzed by GLC (Shimadzu model GC-14B, Kyoto, Japan) fitted with a 2 m × 1/8″ stainless steel column packed with GP 3–2310/2% SP-2300 on 100/120 mesh Chromosorb WAW. Injector and detector temperatures were 230°C. Column oven was operated at 200°C, isothermal. Nitrogen carrier gas flow rate was 50 mL/min. Individual peaks of fatty acid methyl esters were determined with a C-R6A Chromatopack integrator (13).

Triglyceride composition was determined by HPLC (14). The HPLC system used was equipped with Shimadzu LC-10 AD liquid chromatograph, Shimadzu SIL-10A autoinjector, Shimadzu system controller SCL-10A, and RID-6A Shimadzu refractive index (RI) detector. The column used was a (Milford, MA) Nova-Pak C_{18} (3.9 × 300 mm) Waters packed with a particle size of 5-µm. The mobile phase was a mixture of acetone/acetonitrile (63.5:36.5) and the flow rate was 1 mL/min. The injection volume was 10 µL of 5% (wt/vol) oil in chloroform. Sensitivity was adjusted to 16×10^4 RI units full-scale deflection. TG peaks were identified based on the retention time of TG standards and results of Swe *et al.* (14), Ghazali *et al.* (15), and Wong (16).

A Perkin-Elmer Model DSC-7 differential scanning calorimeter (DSC) (Norwalk, CT) was used for thermal analysis of the samples. The instrument was calibrated with indium and dodecane. The atmospheric condition in the DSC was 1 atm. Gas used for the dry box was pure N_2 (99.99%) with flow rate of 30 psi. Samples of *ca*. 3–10 mg were weighed into aluminum pans, and covers were crimped into place. An empty, covered pan was used as a reference. Both were placed in the instrument sample chamber. Heating and cooling cycles were made on each sample. Samples were subjected to the following temperature program: 80°C isotherm for 5 min, and cooled from 80 to –80°C at a rate of 5°C/min, hold at –80°C for 5 min. The same sample was then heated from –80 to 80°C with rate of 5°C/min (9). Heating and cooling thermogram traces were recorded and maxima and minima peak temperatures, and onset peak positions were tabulated.

RESULTS AND DISCUSSION

Chemical properties. The fatty acid compositions of CPO and its products are shown in Table 1. RBD palm olein and superolein had a higher content of unsaturated fatty acid than CPO, while RBD palm stearin had a higher saturated fatty acid content. These fatty acids are distributed in the various glyceride molecules as shown in Table 2. Swe *et al.* (14) found that the peaks appearing for RBD-palm olein had the following retention time order: LaLaLa, MMLa, LaLaM, MPL, MPO, MMM, LLL, OOL, PLO, PPL, OOO, POO, PPO, OOS, POS, SLS, SOS, while Ghazali *et al.* (15) found that the peak which appeared between PPO and OOS was

TABLE 1
Fatty Acid Composition of CPO, RBD Palm Oil, RBD Palm Olein, RBD Palm Stearin, and Superolein

	Fatty acid composition (%)				
Fatty acid	CPO[a]	RBD palm oil	RBD olein	RBD stearin	Superolein
Saturated					
M	0.93	0.92	0.89	1.21	0.81
P	45.48	46.30	41.54	61.21	38.47
S	3.49	3.52	3.51	4.00	3.14
Total	49.91	50.74	45.94	66.42	42.42
Unsaturated					
O	40.17	39.58	43.63	27.54	45.77
L	9.92	9.68	10.43	6.05	11.81
Total	50.09	49.26	54.06	33.59	57.58

[a]CPO, crude palm oil; RBD, refined, bleached, deodorized; M, myristic; P, palmitic; S, stearic; O, oleic; L, linoleic.

TABLE 2
Glyceride Composition of CPO, RBD Palm Oil, RBD Palm Olein, RBD Palm Stearin, and Superolein

	Glyceride composition (%)				
Fatty acid	CPO[a]	RBD palm oil	RBD olein	RBD stearin	superolein
Diglyceride	6.32	5.20	5.55	5.15	6.24
Triglyceride	93.60	94.80	94.45	94.85	93.76
Triunsaturated					
OOO	3.90	4.40	4.61	2.14	5.25
OOL	1.22	0.58	0.66	1.81	0.77
Total	5.12	4.98	5.27	3.95	6.02
Monsaturated					
PLO	10.02	9.68	10.63	4.53	12.56
POO	21.39	23.26	25.60	9.40	29.13
OOS	2.78	2.24	2.58	2.47	3.17
Total	34.1	35.18	38.81	16.40	44.86
Disaturated					
MPL	3.03	2.20	2.52	2.22	2.99
PPL	9.37	9.23	9.61	7.18	10.14
PPO	27.39	29.62	29.64	23.36	22.46
POS	5.29	4.90	5.11	3.85	3.97
SOS	1.36	—	0.68	—	0.51
Total	46.43	45.95	47.56	36.61	40.07
Trisaturated					
MMM	0.76	0.42	0.46	0.93	0.54
MMP	2.38	1.70	1.85	2.05	2.27
PPP	4.81	5.51	0.50	27.16	—
PPS	—	1.06	—	5.06	—
Total	7.95	8.69	2.81	35.20	2.81
Unknown	—	—	—	2.69	—

[a]See Table 1 for abbreviations. Retention time of unknown peak lies between PPL and OOO.

PPP, and according to Wong (16), the peak which appeared between POS and SOS was PPS (La, lauric; M, myristic; P, palmitic; O, oleic; L, linoleic; and S, stearic acids). Based on these data, the TG composition of CPO was 7.95% trisaturated (MMM, MMP, PPP, PPS), 46.43% disaturated (MPL, PPL, OPP, POS, SOS), 34.1% monosaturated (PLO, OOP, OOS), and 5.12% triunsaturated (OOL, OOO). TG compositions of CPO and RBD palm oil were quite similar. These results agree with the results of Ong *et al.* (2). Palm olein contains more disaturated and less trisaturated TG, while palm stearin contains more trisaturated and less monosaturated TG. Superolein, a product of double fractionation, contains more triunsaturated and monosaturated, and less trisaturated TG. The composition of fatty acids attached to a TG molecule determines the thermal behavior of the TG. The more saturated fatty acids attached, the faster the TG crystallizes. Fractionation separated the TG based on their crystallization point. During fractionation of CPO or RBD palm oil, the stearin that contains more trisaturated TG will crystallize, while the olein that contains less trisaturated TG will remain liquid (Table 2).

Thermal profiles. Heating and cooling thermograms of triglyceride standards are presented in Figures 1 and 2, respectively. Cooling and heating thermograms of CPO, RBD palm oil, RBD palm olein, RBD palm stearin, and superolein are presented in Figures 3 and 4. The CPO and RBD palm oil have five exothermic peaks in cooling thermograms, while in the heating thermograms there are seven endothermic peaks. The peaks of these endothermic and exothermic thermograms were classified into a high-melting (high-T) peak group and a low-melting (low-T) peak group. Cooling and heating thermograms of CPO and RBD palm oil were quite similar, with only slight differences in their temperature of the endothermic peaks.

Heating thermograms—triglyceride standards. In the heating thermograms, a fully saturated glyceride (PPP, MMM) showed a single peak at a high-T, while MMP showed two peaks of which the higher was at 45.12°C. The fully unsaturated glyceride (OOO) also showed a single peak at low-T, and monosaturated glyceride (OOP) showed two peaks of which the higher was at 15.05°C. The disaturated TG (SOS, POS, OPP) showed a single peak between those temperatures (Table 3).

Heating thermograms—palm oil and its products. The heating thermograms of RBD palm olein were found to be different from those of RBD palm stearin, CPO, and RBD palm oil. From the heating thermograms, it seems that it is more difficult to identify the peak temperatures because they overlap one another. For identification of the DSC profile, it is easier to use cooling thermograms rather than heating ones, due to the clear separation of their peaks. This observation agrees with Ng (17) and Burger and Akehurst (18). For lauric oil, however, Rossell (7) found that it was easier to use the heating thermogram.

From the heating thermograms the polymorphic forms of the samples could be deduced. Garti *et al.* (19) used the DSC technique to study the role of TG mixtures on the stabilization of the β′ form. They indicated that the first peak of the heating thermogram corresponds to the melting of the α form, while

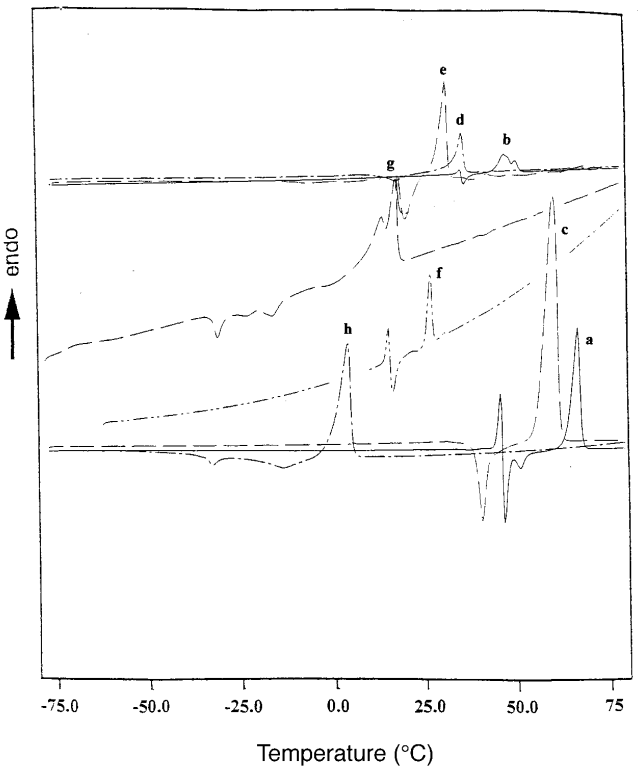

FIG. 1. Melting thermograms of triglyceride standards (a) PPP, (b) MMP, (c) MMM, (d) SOS, (e) POS, (f) OPP, (g) OOP, and (h) OOO run at –80 to 80°C with melting and cooling rates of 5°C/min. P, palmitic; M, myristic; S, stearic; O, oleic.

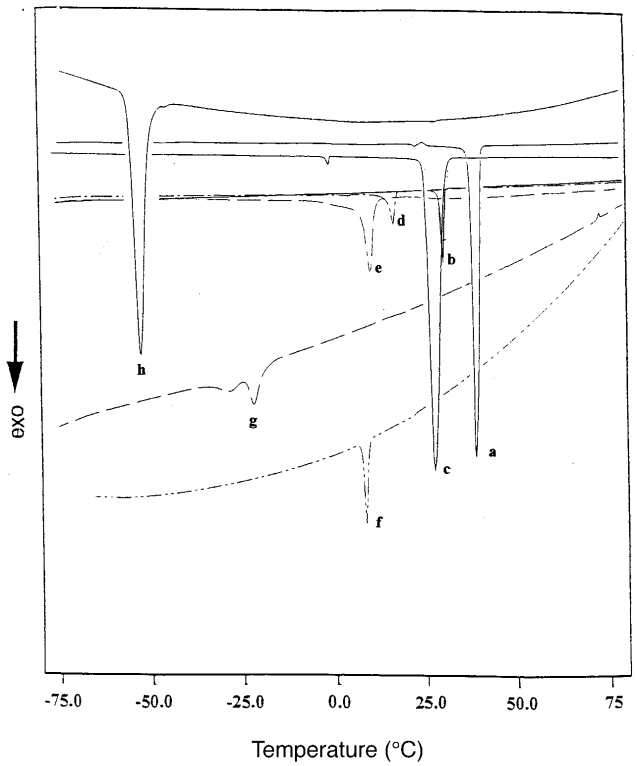

FIG. 2. Cooling thermograms of triglyceride standards (a) PPP, (b) MMP, (c) MMM, (d) SOS, (e) POS, (f) OPP, (g) OOP, and (h) OOO run at –80 to 80°C with melting and cooling rates of 5°C/min. See Figure 1 for abbreviations.

TABLE 3
Temperature of the Peaks of Triglyceride Standards (°C) on DSC Curve for Heating and Cooling in Figures 1 and 2[a]

DSC thermogram		PPP[a]	MMP	MMM	SOS	POS	OPP	OOP	OOO
Heating	High-T	64.55	48.05	58.32	33.38	28.98	25.32	—	—
		—	45.12	—	—	—	—	—	—
	Low-T	—	—	—	—	—	—	15.05	2.98
		—	—	—	—	—	—	12.12	3.05
Cooling	High-T	38.28	29.11	27.64	15.54	8.95	7.84	—	—
	Low-T	—	—	—	—	—	—	–22.22	–52.66
								–28.09	—

[a]DSC, differential scanning calorimeter; High-T, high-melting peak group; Low-T, low-melting peak group. For other abbreviations see Table 1.

the last peak corresponds to the melting of the β form. The intermediate peak indicates the melting of the β′ form. According to Che Man and Swe (9), the heating curves of RBD palm oil showed that the low-temperature peaks represent polymorphs β'_2 and α, while the high-temperature peaks represent polymorphs β'_1 and β_1. Busfield and Proschogo (3) found from the heating thermogram that the structures of palm stearin were α, β′, and β. Based on these studies and our earlier observation using the X-ray diffraction technique (10), we estimated the polymorphic forms of CPO, RBD palm oil, RBD palm olein, RBD palm stearin, and superolein as shown in Table 4.

If the peak temperatures of CPO and RBD palm oil heating thermograms are compared (Fig. 3a and 3b), it is seen that after being refined, bleached, and deodorized, the β'_2 and α forms were bigger in RBD palm oil than in CPO, and the peak height of β'_2 increased while α decreased. The temperature of α peak shifted from 4.78 to 6.25°C. The shifting of these peaks might be due to the refining process, including the use of high-temperature deodorization (260°C). The appearance of β′ and β in RBD palm olein was hardly detectable (Fig. 3). However, RBD palm stearin (Fig. 3) has a very strong peak in β form, and still indicated the presence of olein.

Cooling thermograms—triglyceride standards. In the cooling thermograms, a fully saturated glyceride (PPP, MMP, MMM) showed a single peak at a high-T, a fully unsaturated glyceride (OOO) showed a single peak at low-T, and monosaturated glyceride (OOP) showed two peaks at low-T of which the higher was at –22.22°C. The disaturated TG (SOS, POS, OPP) showed a single peak between those of fully saturated glyceride and OOP (Table 3).

Cooling thermograms—palm oil and its products. The profiles for cooling thermograms of RBD palm olein and superolein were found to be different from those of RBD palm stearin, CPO, and RBD palm oil. The cooling thermograms of RBD palm olein and superolein showed only low-T (T1, T2, T3) peaks whereas the others have both high-T and low-T peaks (Fig. 4). The temperatures of each peak from all samples are shown in Table 4. From the cooling thermogram of CPO, the temperature first onset at 16.64°C and its offset at 7.84°C. This temperature shifted to 17.75 and 10.04°C, respectively, after being refined, bleached, and deodorized as shown in the RBD palm oil cooling thermogram. Ng and Oh (20,21) conducted a repeated crystallization of stearin. At each cycle of recrystallization, its cooling thermogram showed that the high-T peak intensified while the low-T peak diminished in intensity. After five recrystallizations, the low-T peaks disappeared. Therefore, we might conclude that the low-T peak group represents the olein whereas the high-T peak group represents the stearin.

Based on TG composition, stearin contains more disaturated and trisaturated and less monosaturated and triunsaturated glyceride. This stearin fraction is expressed in the high-T

TABLE 4
Temperature of the Peaks of CPO, RBD Palm Oil, RBD Palm Olein, RBD Palm Stearin, and Superolein (°C)[a] on Differential Scanning Calorimeter Curves for Heating and Cooling in Figures 3 and 4

DSC thermogram		Figure[b] a	b	c	d	e	Polymorphic form
Heating	High-T	34.48	36.32	—	55.75	—	β_1
		19.45	21.65	—	39.25	—	β_1'
	Low-T	4.78	6.25	8.45	6.98	8.08	α
		—	4.05	—	—	—	α′
		–1.08	1.12	3.32	2.58	4.41	β_2'
		–6.22	–6.22	–5.75	—	–5.85	—
		–19.42	–17.95	–16.48	—	–15.02	—
Cooling	High-T	13.72	15.54	—	27.64	—	β_1'
	Low-T	–3.89	–2.42	–1.69	–1.69	–4.62	α
		–11.22	–10.86	–8.65	—	—	β_2'
		–25.89	–26.36	–29.19	—	–28.09	—
		–51.55	–48.62	–60.36	—	–58.89	—

[a]Values given are read from right to left.
[b]Referring to Figures 3 and 4, a = CPO, b = RBD palm oil, c = RBD palm olein, d = RBD palm stearin, e = superolein.

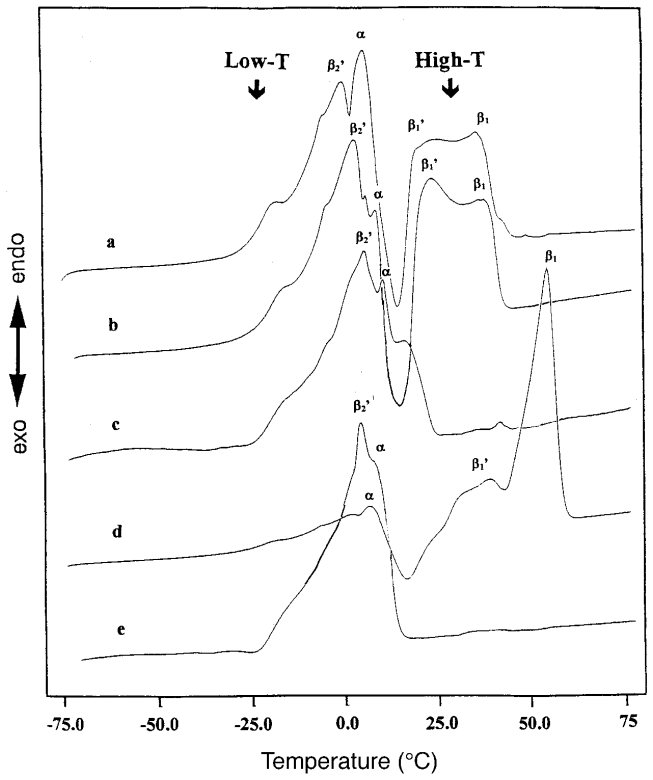

FIG. 3. Melting thermograms of (a) crude palm oil (CPO); (b) refined, bleached, deodorized (RBD) palm oil; (c) RBD palm olein; (d) RBD palm stearin; (e) superolein run at –80 to 80°C with melting rates of 5°C/min.

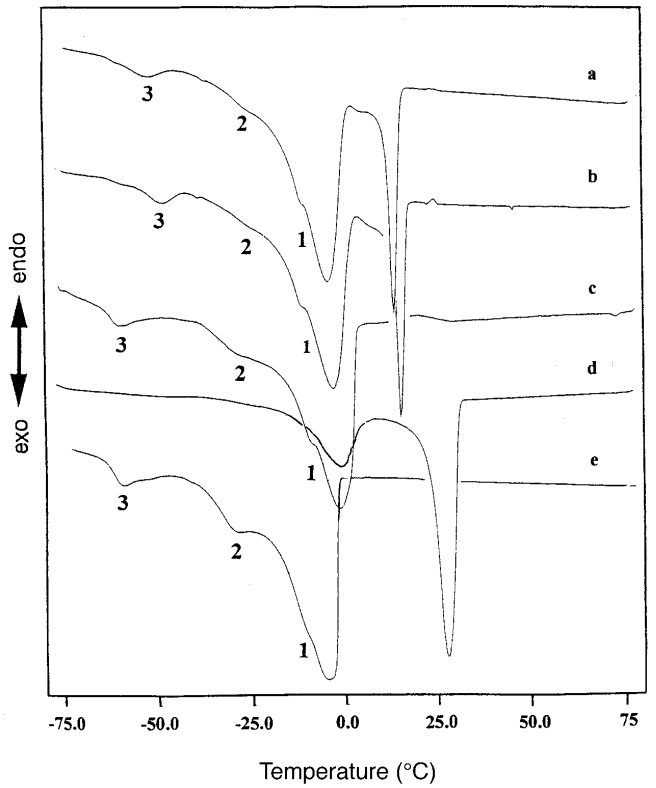

FIG. 4. Cooling thermogram of (a) CPO, (b) RBD palm oil, (c) RBD palm olein, (d) RBD palm stearin, (e) superolein run at –80 to 80°C with cooling rates of 5°C/min. See Figure 3 for abbreviations.

peak of the DSC thermogram. The olein, on the other hand, contains more monosaturated, disaturated, and triunsaturated, and less trisaturated glyceride. This olein fraction is expressed in the low-T peak of the DSC thermogram. In the DSC thermogram of olein, the high-T peaks did not appear, while for the CPO, RBD palm oil, and stearin they did (Table 4). Based on TG composition, the olein has low trisaturated TG (2.81%), CPO and RBD palm oil have 7.95 and 8.69%, while stearin has 35.20%. Therefore, it can be concluded that the high-T peaks reflected the presence of the trisaturated TG (Figs. 3,4). These results agree with Wong (16).

The cooling thermogram of superolein showed higher peaks at the temperature ranges of −48.62 to −60.36°C and −25.89 to −29.19°C compared to that of olein fraction, while the peak at range of −11.22 to −1.6°C in superolein is much lower than that in olein (Fig. 4). Superolein contains higher triunsaturated and monosaturated TG and lower disaturated TG than the in olein fraction. The cooling thermogram of the triunsaturated (OOO) standard has a single peak at −52.66°C, whereas monosaturated (OOP) is at −22.22°C (Fig. 2, Table 3). Therefore, it is confirmed that the peak at the temperature range of –48.62 to –60.36°C (low-T3) in either superolein or olein is the triunsaturated TG (OOO), the peak at the temperature range of −25.89 to −29.19°C (low-T2) is the mixture of disaturated TG (PLO, POO, OOS), and the peak between −11.22 and −1.69°C (low-T1) is the mixture of monosaturated TG (MPL, PPL, PPO, POS, SOS) (Figs. 3,4).

The thermogram of the stearin showed a small low-T group peak, which indicates that the stearin still contains some occluded olein. This means the fractionation that produced this olein was not optimal. This study showed that DSC can be used to determine the thermal properties of palm oil products at various stages in the fractionation plant. The thermograms of the input material are very useful as a reference for determining the appropriate fractionation conditions. The thermograms of the product are also important to determine the success of a fractionation process. TG composition of palm oil can be obtained by HPLC analysis, however this method does not reflect the thermal profile. Although DSC did not display the individual TG as HPLC did, it displayed the TG group as triunsaturated, trisaturated, disaturated, and monosaturated TG, which is very meaningful for fractionation because the process is basically aimed at separating the liquid fraction, which is a group of unsaturated TG, from the solid fraction, which is a group of saturated TG. Therefore, the relationship between the TG composition and DSC thermal profile of palm oil and its products has the potential to be utilized as a quality control method in the fractionation plant.

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