A Study on the Crystal Structure of Palm Oil-Based Whipping Cream

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ABSTRACT: The crystal structure of palm oil-based whipping cream was studied. Various proportions of refined, bleached, and deodorized palm oil (RBDPO) and palm kernel oil (RBDPKO) were prepared and their crystal structure and thermal profile were observed by an X-ray diffractometer and a differential scanning calorimeter, respectively. The results showed that all blends possessed a stable and shiny β′ polymorph at 25°C, which showed that RBDPO and RBDPKO were appropriate for producing a nondairy whipping cream. Some eutectic effect was observed in some blends, which was attributed to the complex combination of TAG in RBDPO and RBDPKO.

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KEY WORDS: Differential scanning calorimetry (DSC), nondairy whipping cream, RBDPO, RBDPKO, X-ray diffraction (XRD).

The fats and oils present in natural plant sources are mixtures of different types of TAG. The complicated behavior of melting, crystallization and transformation, crystal morphology, and aggregation of real-fat systems is due in part to the physical properties of the component TAG and in part, or more importantly, to the phase behavior of the mixtures. A TAG usually possesses three polymorphs: α, β′, and β. The crystallization behavior of the TAG, such as crystallization rate, crystal size and crystal network, crystal morphology, and crystallinity, is directly influenced by polymorphism, whereas polymorphism *per se* is influenced by several external factors, such as temperature, pressure, solvent, rate of crystallization, and impurities (1).

The crystallization behavior of fats and lipids plays an important role in two major industrial processes: (i) processing of the end products made from fat crystals, such as chocolates, whipping creams, and so on, and (ii) separation of specific fats and lipids from the natural plant sources (1). The amount and type of crystals (or solids) present and the strength of the crystal network determine the texture. β′ crystals are the most desirable because they are relatively small and can incorporate a large amount of liquid oil into the crystal network (2). At least 40% of the fat must be crystalline to promote partial coalescence and add rigidity to the air bubble interface (3).

Temperature strongly influences the physical properties of fat crystals. Fats can undergo a reversible solid–liquid phase transition (4), which can exert an influence on the final rheological behavior of an emulsion (5).

The presence of fat crystals in whipping cream can either stabilize or destabilize emulsion droplets, depending on whether they are intraglobular or in the continuous phase (6). Rousseau (7) investigated the properties of fat crystals and their influence on emulsion stability and found that crystals had a determining impact on the structure and stability of an emulsion. To assist in the stabilization of emulsions, fat particles (such as fat crystals) must collect at the emulsion droplet interface, providing a physical barrier to coalescence. Emulsifiers can also interact with fat crystals. Proteins play a dominant role in the formation of the foam because they orient around the air cells and stabilize the foam (8). For example, lecithin has been shown to adsorb to fat crystals, making their surface more polar (9).

Palm oil, which is rich in β′ crystals, is a complex, multicomponent mixture of different TAG with different m.p. The unique crystallization behavior of palm oil is caused by the composition of the TAG and related compounds, e.g., FA and partial acylglycerols (10).

The use of X-ray diffractometry (XRD) is the most direct technique for studying polymorphism and can be the most informative. An XRD pattern comprises both long- and short-spacings. The short-spacings are related to the cross-sectional arrangement of the crystal lattice and are particularly independent of the chain length of FA (11). DSC is the most widely used thermoanalytical technique for studying oils and fats. This technique is used for studying various heat-related phenomena in materials by monitoring associated changes in enthalpy and monitoring the phase behavior of TAG mixtures (12). In this study, DSC and XRD were used to study the crystal structure of palm oil-based whipping creams.

MATERIALS AND METHODS

Materials. Refined, bleached, and deodorized palm kernel oil (RBDPKO) and refined, bleached, and deodorized palm oil (RBDPO), sucrose, corn syrup solids, sodium caseinate, soybean lecithin, carboxymethylcellulose, and MAG were provided by the Malaysian Palm Oil Board.

Methods. RBDPKO and RBDPO were kept overnight at 60°C and then blended. The proportion of RBDPO/RBDPKO for 11 blends was: B1 = 100:0, B2 = 90:10, B3 = 80:20, B4 = 70:30, B5 = 60:40, B6 = 50:50, B7 = 40:60, B8 = 30:70, B9 = 20:80, B10 = 10:90, and B11 = 0:100. The oils were then mixed

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with the remaining ingredients, made into emulsions, and whipped as described previously by Shamsi *et al.* (13).

(i) Determination of slip melting point (SMP). The SMP for the blends was determined according to PORIM test methods (14). Four capillary tubes (o.d. 1.4–1.7 mm, i.d. 1.1–1.3 mm, length 50–60 mm) were lowered into each blend so that the oil, which was kept at 60°C overnight, rose 10 mm in the tubes. The capillary tubes were then rolled against a piece of ice to solidify the oil to fat. The capillary tubes were held in test tubes in a beaker full of ice water for 1 h in the refrigerator. After 1 h the capillary tubes and a thermometer were fastened to glass rods in a beaker containing 600 mL distilled water and cooled to 5°C. A magnetic stirrer was used to stir the water. Heat was then applied using a heating coil element by turning it on for 10 s and off for 50 s so that the temperature rose 1°C per min. The temperature was recorded when the fat changed to oil and began to rise in the capillary tubes. The experiment was carried out in duplicate; the mean for the two experiments was calculated and considered the SMP for each tube.

(ii) Determination of solid fat content (SFC). The SFC for the blends was measured according to PORIM test methods (14) using a Bruker NMS 120 minispec nuclear magnetic resonance analyzer (NMRA) (Rheinstetten, Germany). A 1.7-g portion of each sample was poured into nine test tubes, and the tubes were then placed in a water bath at 70°C for 30 min. The nine tubes were next chilled to a temperature of 0°C for 90 min, and then transferred to temperatures of 5, 10, 15, 20, 25, 30, 35, 37 and 40°C, respectively, for 90 min. The tubes were finally transferred to the NMRA.

(iii) Thermal analysis of blended oils by DSC. A Perkin-Elmer differential scanning calorimeter, model DSC-7, equipped with a thermal analysis data station (PerkinElmer Corp., Norwalk, CT) was used. Nitrogen (99.999% purity) was used as the purge gas, at a flow rate of approximately 20 mL/min. The DSC instrument was calibrated using indium and *n*-dodecane. Samples of 4–15 mg were weighed into aluminum pans to the nearest 0.1 mg, and covers were hermetically sealed into place. An empty, hermetically sealed aluminum pan was used as reference prior to the analysis of samples. Samples were subjected to the following temperatures: 80°C isotherm for 5 min, cooled at 10°C/min to −80°C, held for 5 min, and then heated to 80°C at 5°C/min. The manufacturer's software program (7 Series UNIX DSC software library) was used to analyze and plot the thermal data as described previously (12).

(iv) XRD. The polymorphic form of the fat crystals in the samples was determined as described by AOCS Method Cj 2-95 (15). An FT 592 Enraf-Nonius Diffractis X-ray generator (Delft, The Netherlands) and an Enraf-Nonius Model FR 552 Guinier camera were used. The samples were kept at 25°C for 24 h to stabilize them. The customized single-compartment cell, equipped with a temperature control by means of an external-circulation thermostated bath, was set at 25°C and the crystal networks of the samples were observed. Kodak (Eastman Kodak Co., Rochester, NY) direct-exposure diagnostic film was used, and the spacings on the X-ray film were measured with an Enraf-Nonius Guinier viewer capable of reading to the nearest 0.001 nm under illuminated magnification.

RESULTS AND DISCUSSION

Regardless of the ratio of RBDPO to RBDPKO, all blends possessed the β′ polymorph at 25°C, which produced a shiny and stable foam (Table 1). When air incorporation is considered important, as in whipping cream, β′ crystals provide the cream with many small bubbles and greater volume, whereas β crystals allow the incorporation of relatively little air (16).

The α short spacings are found at 4.15, 4.2, and 3.8 Å; β are found at 4.6, 3.8, and 3.7 Å; and β′ are found at 4.2 and 3.8 Å (17). Levels of β and β′ crystals in mixtures are estimated by the relative intensity of the short-spacings of 4.2 and 4.6 Å (18). The short-spacings in the whipping creams in this study were between 4.50 and 3.80 Å, which is a distinctive pattern for β′ crystals. The creams were stable and showed no syneresis at 25°C (13). β′ crystals are small and needle-like and grow together to produce a network. One important feature of this crystal network is the very large overall crystal surface, which enables the liquid oil and water droplets to be retained (16). This can be considered an advantage for the application of RBDPO and RBDPKO when producing nondairy whipping cream. However, it should be considered that although all blends possessed the β′ polymorph, not all were suitable for producing a whipping cream.

SFC is an important criterion for a fat or oil to be applied in a nondairy whipping cream. Blends B7 to B11 should leave no greasy aftertaste in the mouth since their SMP ranged from 26.25 to 27.5°C and the SFC was less than zero as well (Tables 2 and 3). By increasing the temperature to 40°C, the syneresis in creams was limited to only a few drops (13). The fats with SMP below body temperature produce a pleasant cooling effect in the mouth when consumed (19).

When fats of different compositions are mixed, they may show a eutectic effect. This means that the m.p. or SFC for the blend is lower than that of the individual components (20). This phenomenon was observed in blends B8 and B9 (Table 2). The m.p. for these two blends, measured by DSC, was lower than

TABLE 1
X-ray Diffraction Patterns of 11 Blends of RBDPO[a] and RBDPKO[b] Measured by X-ray Diffractometry After Stabilization at 25°C for 24 h

	Short-spacings (Å)					
Blends	4.5	4.30	4.20	4.00	3.80	PF[c]
B1		4.34 m[d]	4.20 s[e]	4.02 s	3.84 s	β′
B2		4.34 m	4.20 s	4.02 s	3.84 s	β′
B3		4.34 m	4.20 s	4.02 s	3.84 s	β′
B4		4.34 m	4.20 s	4.02 s	3.84 s	β′
B5	4.58 w	4.34 m	4.20 s	4.02 s	3.84 s	β′
B6	4.58 m	4.34 w[f]	4.20 s	4.02 s	3.84 s	β′
B7	4.58 w	4.40 vw[g]	4.23 m	4.06 w	3.82 m	β′
B8		4.40 vw	4.23 m	4.06 w	3.82 m	β′
B9		4.40 vw	4.23 m	4.06 w	3.82 m	β′
B10		4.40 vw	4.23 m	4.06 w	3.82m	β′
B11		4.37 m	4.23 s	4.04 w	3.82 s	β′

[a] Refined, bleached, and deodorized palm oil.
[b] Refined, bleached, and deodorized palm kernel oil.
[c] PF, polymorphic form.
[d] m, medium.
[e] s, strong.
[f] w, weak.
[g] vs, very weak.

TABLE 2
Crystallization and m.p. of Various Blends of RBDPKO and RBDPO Measured by DSC

Samples	Crystallization temperature (°C)	Crystallization onset (°C)	m.p.[a] (°C)	Onset of m.p. (°C)	SMP (°C)
B1	3.82	21.12	43.47	−10.81	37.5 ± 0.7
B2	2.49	19.79	42.07	−13.47	36.15 ± 1.2
B3	−8.48	18.79	41.74	−13.47	34 ± 1.41
B4	−4.82	17.46	40.08	−13.8	31.5 ± 2.12
B5	−1.49	16.79	39.41	−7.15	32.25 ± 0.35
B6	−2.16	16.13	38.08	2.49	30.75 ± 2.12
B7	−4.49	14.8	37.74	−0.83	27.5 ± 0.7
B8	−6.15	14.8	28.1	−5.82	26.9 ± 0.14
B9	−3.82	15.46	28.77	−6.15	26.25 ± 0.35
B10	0.4	9.14	31.43	−11.8	26.5 ± 0.7
B11	2.16	8.48	31.43	−12.4	26.85 ± 1.2

[a] Melting point values measured by DSC.
[b] Slip melting point (SMP) values, measured by the PORIM test method (14), represent mean ± SD of duplicate analyses. For other abbreviations see Table 1.

the individual oils of B1 (RBDPO) and B11 (RBDPKO). Because of molecular size differences, RBDPO and RBDPKO are incompatible with each other and form eutectic mixtures (4). The eutectic interaction is clearly shown by the minima in the SFC curves as well (Fig. 1).

B1 to B11 showed a wide variation in crystallization temperatures, from 3.82 to –8.48 (Fig. 2 and Table 2). This variation could be attributed to the complex combination of TAG present in the RBDPKO and RBDPO and also the dominance of RBDPKO. The m.p., measured by DSC, showed a sudden decrease from B7 (37.74°C) to B8 (28.1°C) (Table 2), whereas the SMP, measured by PORIM test methods (14), showed a steady decrease (Fig. 3). Figure 3 shows the difference between the m.p. and SMP measured by DSC and PORIM test methods (14), respectively. The SMP, measured by a standard method, is an "apparent" m.p., whereas DSC has the advantage of showing the range of melting for the whole FA content of the fat or oil rather than a specific point. The m.p., measured by DSC, was capable of identifying eutectic phenomenon in the blends, whereas the SMP, measured by the standard method, showed only a steady decrease (Table 2). The higher percentage of RBDPKO, however, did not decrease the SMP, because only B8 and B9 formed eutectic mixtures. In the standard method of measuring SMP, the values were lower compared to their respective values determined by DSC because the heating rate using DSC was 5°C/min, whereas this rate for the standard method was 1°C/min. However, the *r* in both curves was close (0.81 and 0.90).

Figures 4 and 5 display the cooling and heating thermograms, respectively, of 11 blends of RBDKO and RBDPO. In the cooling thermograms (Figs. 4A–4C) there were two curves, one a high-temperature (HT) curve and the other a low-temperature (LT) curve, represented by A and B, respectively. The HT curve represents the presence of tristearin, whereas the LT curve indicates triolein. In B1, which was pure palm oil, there were two neat curves, whereas in the other blends there were some additional small curves whose appearance could be attributed to the difference in m.p. of each TAG type. Addition of RBDPKO to the RBDPO lowered the crystallization temperature, and the onset of crystallization decreased from 21.12 (B1) to 8.14°C (B11) (Table 2). Addition of RBDPKO reduced the size of peak A, whereas peak B became more pronounced. Since the HT peak (peak A) represented the higher-melting TAG of the blends, the lower m.p. in the blends containing a higher percentage of RBDPKO resulted in a reduction in peak size. The changes observed in crystallization temperature from B1 to B11 could be attributed to the complex combination of TAG of various sorts. Edible oils are complex, multicomponent

TABLE 3
Solid Fat Content of Various Blends of RBDPKO and RBDPO

	Solid fat content[a] (%)										
WBT[b] (°C)	B1	B2	B3	B4	B5	B6	B7	B8	B9	B10	B11
5	65.32	62.57	61.87	61.82	62.64	65.10	65.73	69.07	70.22	72.20	75.17
10	53.45	49.34	46.32	46.15	48.11	50.31	54.83	59.07	61.29	64.98	68.84
15	38.19	34.50	29.21	26.99	28.34	31.53	34.65	39.17	42.83	48.91	55.54
20	23.83	20.58	15.68	12.29	11.55	13.50	17.26	20.64	23.52	30.05	36.46
25	12.85	10.84	8.78	6.46	4.46	3.26	2.66	2.67	4.78	7.98	12.04
30	7.81	6.11	4.82	3.85	2.40	1.22	0.82	−0.91	−1.31	−1.66	−1.59
35	3.97	2.46	1.05	0.12	−0.85	−1.40	−1.78	−1.83	−2.33	−2.15	−2.06
37	2.35	1.02	0.39	−0.49	−1.00	−1.38	−2.76	−2.06	−2.2	−2.26	−2.10
40	1.32	0.05	−0.92	−1.89	−2.00	−2.05	−2.22	−2.16	−2.36	−2.38	−2.42

[a] The minus sign before some values shows the degree of liquidity (e.g., –0.49 shows that the blend is less liquid than –1.89).
[b] WBT, water bath temperature. For other abbreviations see Table 1.

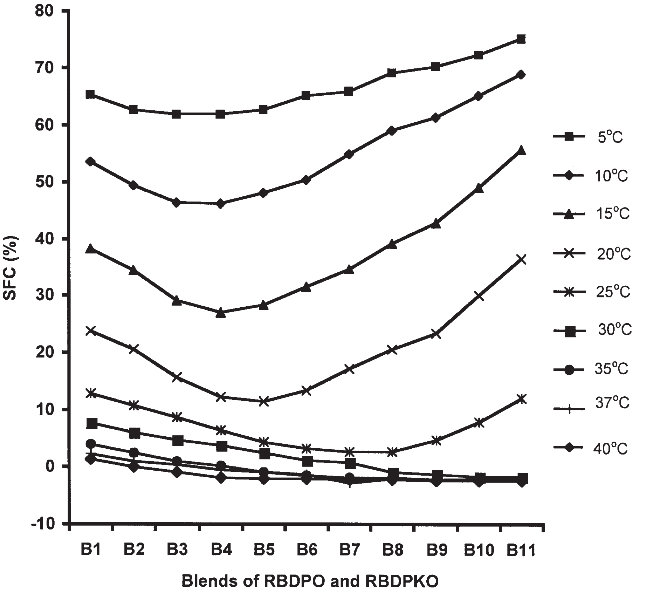

FIG. 1. Solid fat content (SFC) of refined, bleached, and deodorized palm oil (RBDPO) and refined, bleached, and deodorized palm kernel oil (RBDPKO). The key shows the temperatures used to measure the SFC of the whipping cream. See text for description of blends.

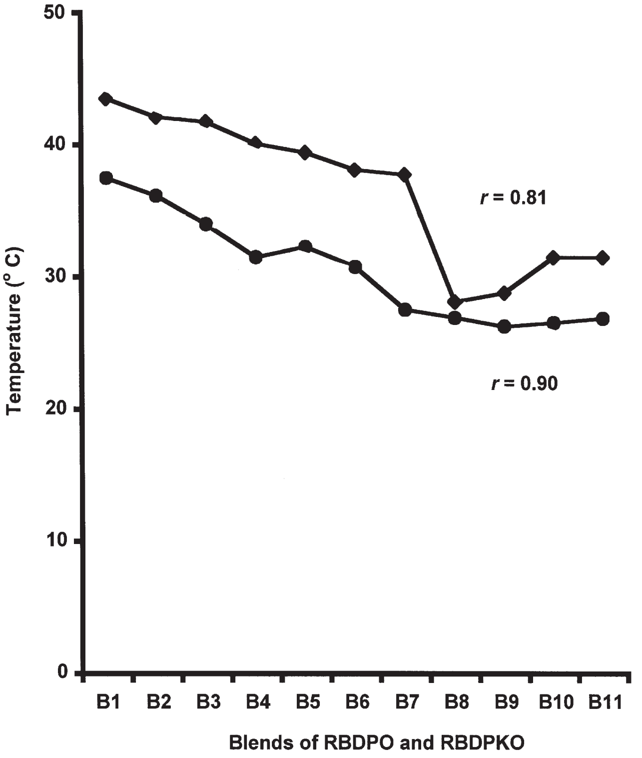

FIG. 3. Slip melting point of the RBDPO and RBDPKO blends measured by DSC (–◆–) and the PORIM test method (–●–) (14). For abbreviations see Figure 1. For blends see text.

mixtures of different TAG with different m.p. (10). The characteristics of the high-melting (HM) glycerides of a fat are important because the TAG composition of the HM glycerides dictates the polymorphic forms in which the fat crystallizes and its future behavior on storage (21). RBDPKO contains a wide range of TAG with m.p. that vary from 72 (SSS, S = stearic) to −10°C (LLL, L = linoleic) (20), which could produce drastic and unexpected changes when blending with RBDPO. The dominant TAG in RBDPKO is LLL (L = lauric), whereas the dominant TAG in RBDPO is POP (P = palmitic; O = oleic) (12), whose m.p. is 34°C (20). The unexpected changes in oil combinations can also be attributed to a eutectic effect, as discussed earlier.

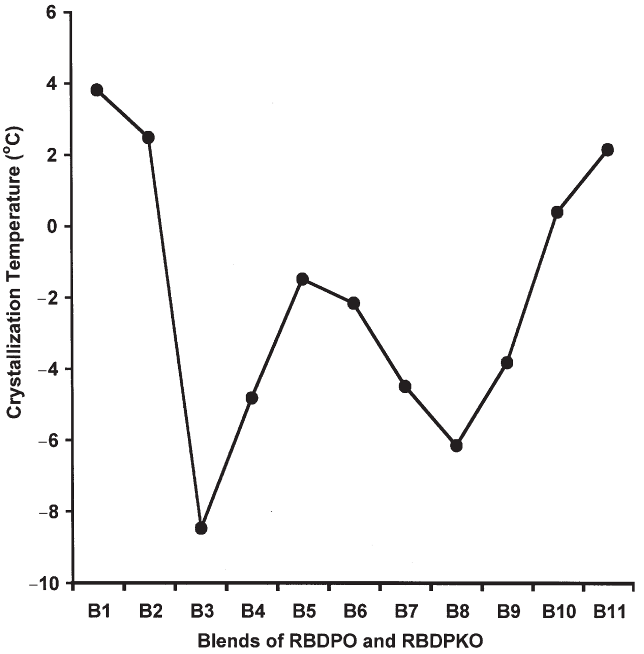

FIG. 2. Variations in the crystallization temperatures of the RBDPO and RBDPKO blends. See text for description of blends. See Figure 1 for abbreviations.

In the heating thermograms shown in Figure 5, the low-melting (LM) and HM curves represent the onset and completion of melting, A and B, respectively. The LM curve represents the m.p. for triolein, which is about 5°C, and the HM curve is the termination point indicating tristearin, whose m.p. is about 72°C. Generally, the heating curves (Fig. 5) showed less sudden changes than the cooling curves (Fig. 4).

DSC was found to be a useful technique for investigating the transition of crystal forms during the melting of fats. When fat is being heated, it can exhibit multiple melting behavior and each recrystallization step represents the transition of a polymorph from its less stable form to a more stable form. Since the more stable crystal form has a higher m.p. than the less stable form, the transition peak temperature can serve as an important indicator of the polymorphic form of the crystals. The β' stabilization of palm oil is most likely due to a decrease in the uniformity of FA (22). The DSC heating curves for B1–B3 (Fig. 5A) and B4 (Fig. 5B) show fusion peaks at 10 and 20°C, indicating that a transition has taken place.

Judging the crystal structure of mixed oils is not an easy task. Any oil or fat has its own physical characteristics,

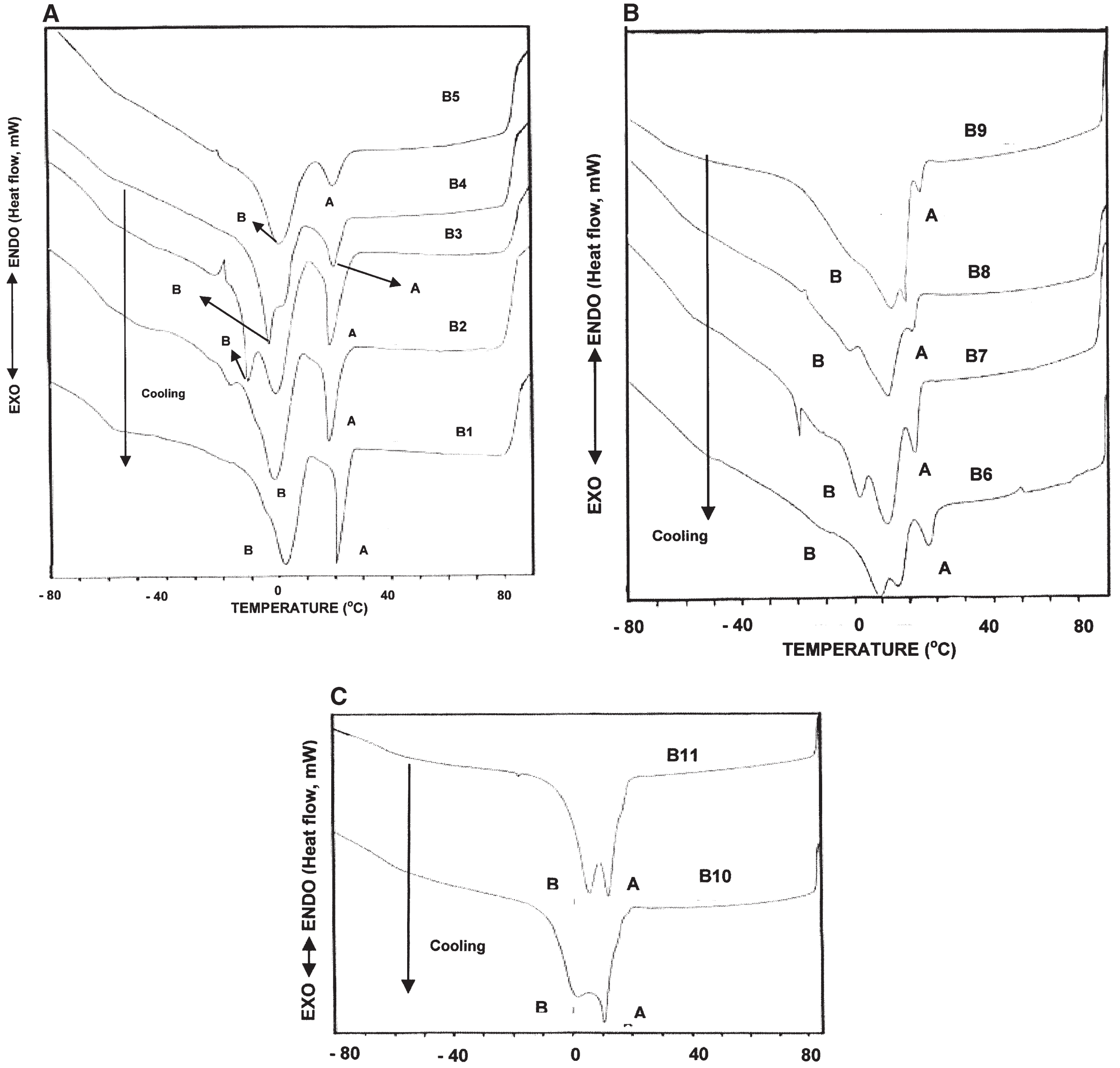

FIG. 4. DSC cooling thermograms of blends of RBDPKO and RBDPO. (A) Blends B1–B5; (B) blends B6–B9; (C) blends B10 and B11. The cooling rate was 10°C/min. Curves A and B show the onset and completion of crystallization, respectively. For abbreviations see Figure 1.

including the m.p. and crystal network, and very few researchers have reported a correlation between DSC and XRD findings for these mixed oils. In addition, investigating the behavior of noninteresterified mixed oils at different temperatures makes the prediction of the results more complicated because of the occurrence of eutectic effects, as in this study. Thus, XRD was carried out for the samples at 25°C only to determine the crystal network. Usually, no α crystals appear at temperatures above 22°C (23); therefore, XRD can be expected to trace α crystals below 22°C in DSC curves. The crystal network undergoes tremendous transformation during the heating or cooling process. When cooling, the α polymorph is produced, which is usually converted to β′ (20). However, at temperatures above 70°C no crystal network can exist (23).

DSC covers a wide thermal range, as in this experiment, from –80 to 80°C. Over this range, the largest number of β′ crystals can be observed at 25°C (Fig. 5A). As can be seen in Table 1, all samples possessed β′ polymorphs, which are far from being as unstable as α.

The m.p. for β′ and β crystals of major TAG in palm oil are 68.8 (PSP, P = palmitic; S = stearic) and 66.2°C (PPP,

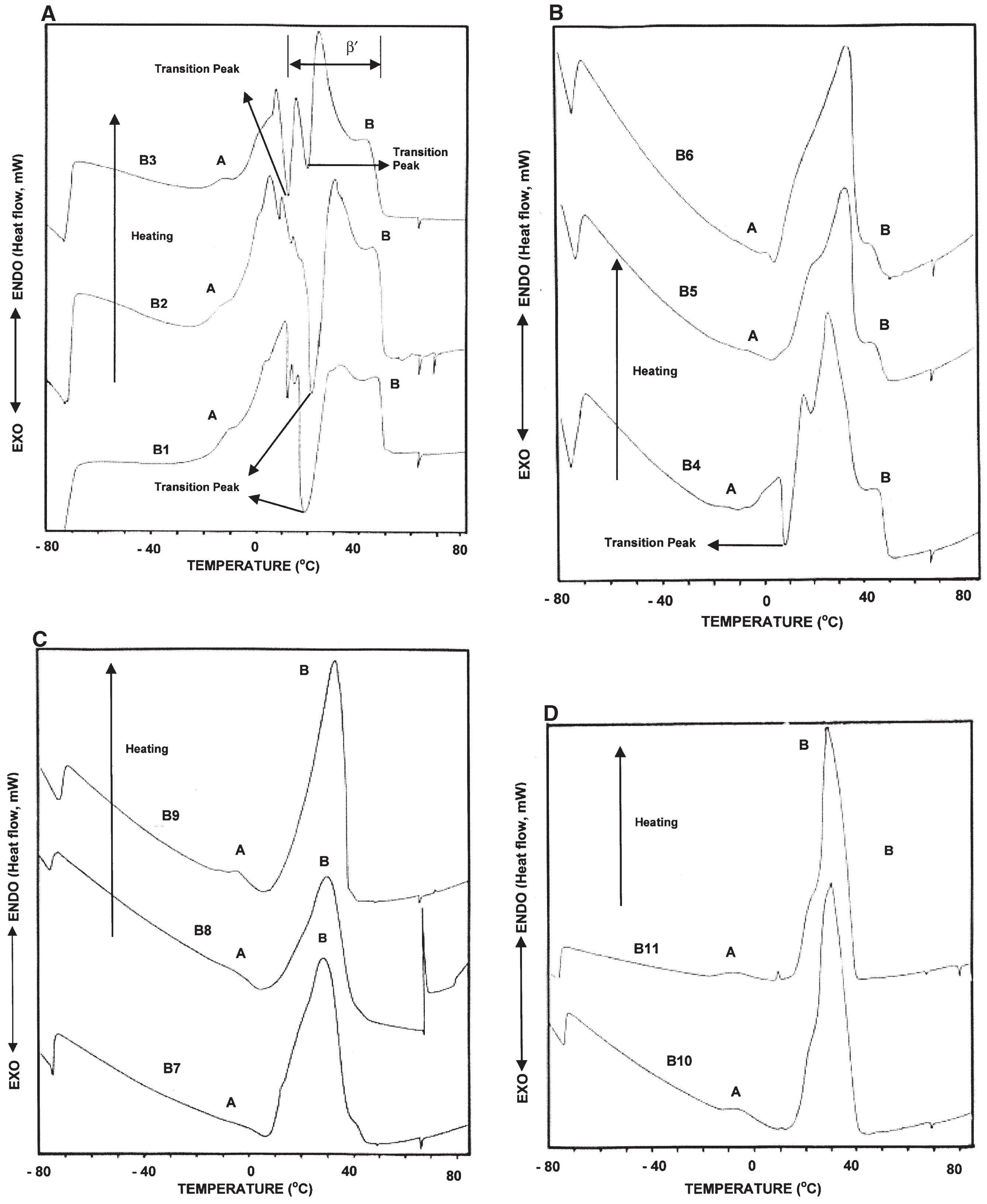

FIG. 5. DSC heating thermograms of blends of RBDPKO and RBDPO. (A) Blends B1–B3; (B) blends B4–B6; (C) blends B7–B9; (D) blends B10 and B11. The heating rate was 5°C/min. Curves A and B show the onset and completion of melting, respectively. For abbreviations see Figure 1.

P = palmitic), respectively (20). Therefore, it is possible to anticipate that the β′ crystals are still present at 25°C, which can preserve the stability of the foam in the palm oil-based whipping cream, as shown in this study (Table 1). Shamsi *et al.* (13), in a study on the stability of foam in whipping creams prepared from RBDPO and RBDPKO blends, found that, after whipping, the samples were still stable at 40°C with only a few drops of serum separated.

The present results showed that all blends possessed a stable β′ polymorph at 25°C, indicating that RBDPO and RBDPKO are suitable for application in a nondairy whipping cream if no solid fat is left below 37°C. A eutectic effect was observed in some blends, which could be attributed to the complex combination of TAG present in RBDPO and RBDPKO.

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