Effect of Emulsion Temperature on Physical Properties of Palm Oil-Based Margarine

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ABSTRACT: Margarines made from refined, bleached, and deodorized palm oil at different emulsion temperatures showed no significant difference in their consistency, polymorphic behavior, and solid fat content (SFC) during storage, although differences were observed during processing. The emulsion temperatures studied were 40, 45, and 50°C, with other parameters such as emulsion flow rates, tube cooler temperature, and pin rotor speed kept constant. The SFC developed during processing and storage at 28°C was measured to evaluate the quality of margarine. The emulsion contained no SFC at any emulsion temperature studied. However, the amount of SFC in the perfector or tube cooler unit increased to 15.9, 13.9, and 15.6% in margarine produced at emulsion temperatures of 40, 45, and 50°C, respectively. At 40°C, the lowest SFC was developed during storage even though this margarine had the highest consistency. The softening point of this sample was moderately high and closely related to the type of crystal developed, which was a mixture of β′ and β crystals. Emulsion at 45°C gave the most stable margarine consistency and SFC with crystal in the β′ form even after the fourth week. At 50°C, moderately soft product was produced, which might be undesirable for some applications, although the crystals were in the β′ form.

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KEY WORDS: Emulsion temperature, palm oil, solid fat content (SFC), tube cooler.

Margarine and table spreads are water-in-oil emulsions. The aqueous phase consists of water, salt, and preservatives (1,2). The oil phase, which contributes to the polymorphic behavior of margarine, is a blend of oils and fats (3–5). Lecithin, distilled MAG, and distilled DAG are common emulsifiers added to the fat phase together with flavoring agents, antioxidants, and coloring agents.

Emulsification is a process in which the water and oil phases are mixed to form a water-in-oil emulsion with the assistance of an emulsifier (1,6–8). Emulsification is a very important step in margarine processing because it ensures the development of the desired crystal form (7). Different types of churns have been used to produce good margarine. These are jacketed with a flow of hot or cold water to allow temperature control of the emulsion. The temperature of the oil phase is maintained at 2–3°C above its m.p., whereas the aqueous phase is held at 7–10°C. After emulsification, the temperature in the churn is lowered to 7–8°C below the m.p. (7) to initiate the development of crystal nuclei. The emulsion is agitated for 10–15 min and, prior to its passing into the tube cooler unit, the temperature is raised to 2–3°C above the melting point. This heating avoids formation of microscopically sized crystals but does not destroy the previously formed nuclei.

Some studies have shown that temperature control during emulsification influences the solidification and plasticizing process and the final texture of the margarine (7,9). However, other studies have considered the emulsification step less critical than chilling (9,10). Borwanker (10) suggested that emulsification should be done at 45–50°C when all the oil is liquid. However, according to Andersen and Williams (7), the optimal emulsification temperature varies with the oil blend and its m.p. The common practice for most margarine manufacturers in Malaysia is to keep the blend at 55–60°C before emulsification and maintain the emulsion tank at 50°C (Foo, T.K., personal communciation; Tan, T.K., personal communication).

Lowering the emulsion temperature to provide precrystallization in the emulsion tanks, as suggested by Andersen and Williams (7), is less practical, and it would be preferable to install an extra tube cooler unit in the system (11). Partial crystallization of margarine emulsion could take place in the tube cooler and be completed before the product leaves the perfector plant after passing through the intermediate crystallizer and pin rotor.

This study was undertaken to determine whether emulsion temperature in the mixing tank has an effect on the physical properties of the final product during storage.

MATERIALS AND METHODS

Materials. Refined, bleached, and deodorized (RBD) palm oil was obtained from a local refinery. The RBD palm oil had a slip melting point (SMP) of 35.1°C, dropping point of 38.6°C, and crystallization temperature of 22°C. Other ingredients included emulsifier (distilled MAG, 90% monoester, SMP 69°C; source: fully hydrogenated palm oil) from Danisco Ingredients (M) Sdn. Bhd (Prai Industrial Estate, Penang, Malaysia), water (filtered municipal water supply), and vacuum-dried salt.

Chemical and physical analysis. SMP by PORIM Test Method (12) was carried out by first dipping three open-ended capillary tubes with internal diameters of 1.1–1.3 mm and external diameters of 1.4–1.7 mm in a filtered liquid oil so that a

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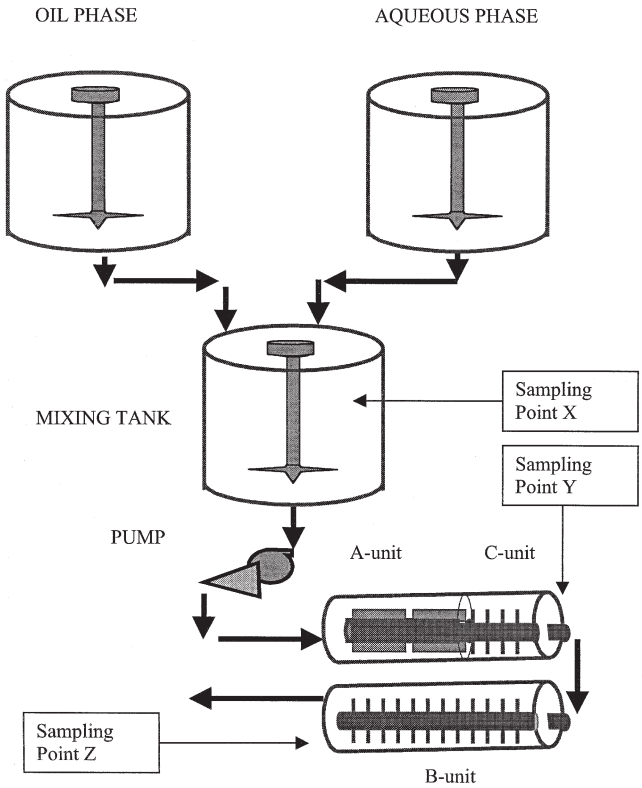
column of fat (~10 mm) was obtained. The tubes were chilled at once in ice, placed in a test tube, and tempered in a 10°C water bath for 16 h. Each tube was tied against a mercury thermometer, dipped in cold distilled water, and heated at 1°C/min until the SMP was reached and the fat in the column rose. Solid fat content (SFC) by PORIM Test Method (13) was determined by pulse NMR (NMS120; Minispec Bruker, Rheinstetten, Silberstreifen, Germany) with reference samples of triolein and tristearin. The oil sample, previously filled in NMR tubes 0.8 cm in diameter to a height of 2 cm, and the reference samples, also in NMR tubes, were melted in a 70°C bath for 30 min. The tubes were cooled at 0°C for 90 min prior to holding for 30 min at the measuring temperature. Measurement was done in sequence at fixed intervals to minimize error.

Production of margarine. A basic recipe that included 80% RBD palm oil, 0.2% emulsifier, 16% water, and 2.5% salt was used for margarine production. Palm oil was melted in a Memmert drying oven (854 UL 80; Schwabach, Germany) at 65°C. The melted oil was weighed into 50-kg production batches and a batch was placed in the mixing tank. Emulsifier, mixed in palm oil in the ratio 1:4 and melted, was added. The water phase containing salt at room temperature (28°C) was then added slowly to the oil phase while agitating it to form a good emulsion. Three experimental batches were made for emulsion temperatures of 40°C (5°C above SMP), 45°C (10°C above SMP), and 50°C (15°C above SMP), and held for 10 min in the mixing tank prior to processing. The margarine was processed in a perfector pilot plant (Gerstenberg and Agger, Copenhagen, Denmark) in the Malaysian Palm Oil Board laboratory. The tube cooler (A-unit) had a volume of 900 mL and a scraped cooling surface area of 0.063 m^2, and attached to it in series was an intermediate crystallizer (C-unit) with a volume of 750 mL. The pin worker (B-unit) had a volume of 3 L. The emulsion was pumped into the A-unit at a throughput rate of 45 kg/h, where it was rapidly cooled. The amount of coolant refrigerating the surface of the tube cooler was set so that the product exiting the tube cooler was 25°C. The scraper blade and the intermediate crystallizer rotated at 250 rpm, whereas the pin worker was 200 rpm.

Sampling procedure. Sampling was carried out at three different places to determine the development of SFC of the emulsion from the mixing tank (sample X), the exit of the scraper unit with intermediate crystallizer (sample Y), and the exit of the pin worker unit (sample Z) (Scheme 1). Duplicate samples were filled into NMR tubes with 0.80-cm diameter and 2-cm height, and the SFC was measured immediately (14). The time between sampling and reading was standardized at 50–60 s to minimize errors. Margarine product was collected in 400-mL tubs at the end of the processing line after the pin worker process for storage studies at 28°C.

Quality assessment. Samples for analysis were placed in a 28°C incubator for 28 d. Consistency of the margarine was determined in terms of penetration yield value (g/cm^2) (15,16), using a cone penetrometer (Stanhope-Seta, Surrey, England) with 40° cone, where the weight of the cone assembly was 79.03 g and penetration time was 5 s. Calculation was



SCHEME 1

according to Haighton (15): $KW/P^{1.6}$, where K = 5840, W = 79.03 + added weight, and P = means of penetration readings. Six readings were taken from each sample every day, and each time a different sample was used.

Isothermal SFC (17) was measured every day, 5 d a week. A solid margarine sample (from the same tub previously used for penetration) was loaded into an NMR tube 0.8 cm in diameter to a height of 2 cm (in duplicates) using a stainless steel piston. The sample was unloaded carefully into the tube to prevent any air space in it. The sample in adiabatic condition was measured by pulsed NMR. Softening point (18) was measured in duplicate on alternate days using a Mettler FP90 (Columbus, OH) at a heating rate of 1°C/min. Polymorphic changes (19) were determined every 5 d using a 601 Diffractis X-ray generator and Guiner X-ray diffraction camera, model FR 552 (Enraf-Nonius, Delft, The Netherlands), operated at 25°C. Data analysis was performed by ANOVA in Microsoft Excel 2000.

RESULTS AND DISCUSSION

Maintaining constant processing conditions while varying the mixing temperature at point X caused the temperatures at points Y and Z (Scheme 1) to differ (Table 1). As the emulsion holding temperature in the mixing tank (point X) was increased, the temperature at point Y decreased. The difference in temperatures at these points was mainly due to the difference in the rate of crystallization. At a holding temperature of 50°C, the difference between the holding and crystallization

TABLE 1
Effect of Emulsion Holding Temperature (point X) on Sample Temperatures at Points Y and Z (see Scheme 1)

Emulsion temperature (°C) (sampling point X)	Product temperature (°C)	
	Sampling point Y	Sampling point Z
40.0	19.0	21.0
45.0	18.0	22.9
50.0	17.7	22.9

temperature of palm oil (22°C) was 28°C. This high temperature difference may have resulted in a long induction period so that little crystallization had occurred by the time the emulsion exited the tube cooler (A-unit) (1). Thus, the temperature measured at point Y was the lowest. In contrast, at a holding temperature of 40°C the emulsion was very close to its dropping point (38.6°C) and the crystallization temperature (22°C) and thus had a shorter induction period. Crystallization occurred in the cooler with a release of heat of crystallization that raised the temperature (1) compared with the sample initially at 50°C.

The sample temperature at point Z, however, was low when the holding temperature was 40°C. As discussed above, only a small portion of the emulsion at holding temperature 50°C was crystallized in the A-unit, although it crystallized more rapidly as soon as it was worked by the pin worker (B-unit). The emulsion at a holding temperature of 40°C, however, crystallized less rapidly in the pin worker unit because crystallization had already started in the A-unit. Thus, the release of heat of crystallization in the sample at an emulsion temperature of 50°C had raised the temperature 1°C higher than at 40°C. A study conducted by Pernille (14) on puff pastry margarine reported a similar observation.

The emulsion in the mixing tank (point X) contained no SFC, because it was held above the SMP of the fats. Thus, an insignificant solid level was detected by the NMR. The SFC of the emulsion increased as it was subjected to shock chilling in the A-unit (Table 2). Sample collected at point Y was in viscous form, indicating a change in its physical structure from liquid to partially crystalline and semisolid (20). However, the SFC of the emulsion was still lower than that for bulk palm oil as shown in Figure 1. Several references suggest that SFC of the fat blend is correlated with the product consistency (1,6); however, our results suggested that the SFC

TABLE 2
Development of Solid Fat Content (SFC) at Different Points During Margarine Production[a]

Emulsion temperature (°C)	SFC (%)[b]		
	Sampling point X	Sampling point Y	Sampling point Z
40.0	0.1 ± 0.0	15.9 ± 0.7	15.7 ± 1.2
45.0	0.0 ± 0.0	13.9 ± 0.1	14.1 ± 0.0
50.0	1.0 ± 0.0	15.6 ± 0.0	15.8 ± 0.7

[a]See Scheme 1 for sampling points X, Y, and Z.
[b]Means of two determinations ± SD.

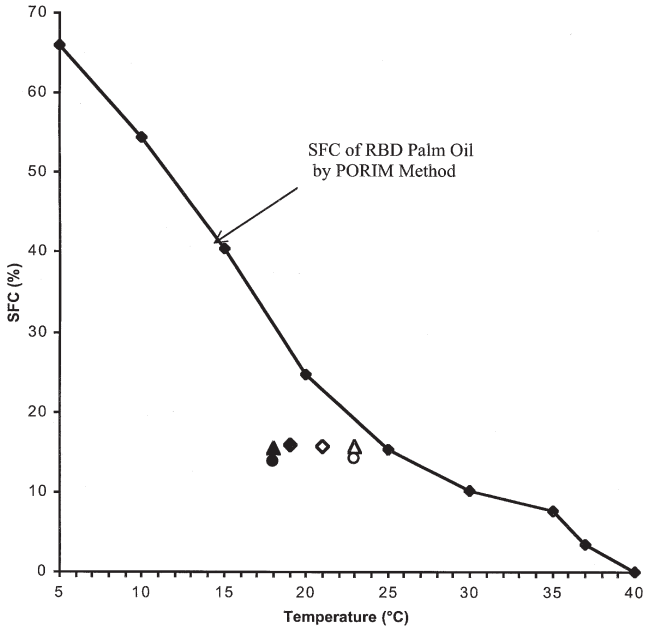


FIG. 1. Solid fat content (SFC) developed at sampling points Y and Z and the SFC of refined, bleached, and deodorized (RBD) palm oil. The SFC of samples at 40°C emulsion at sampling points Y (◆) and Z (◇) were 15.9 and 15.7%, and the product temperatures were 19 and 21°C, respectively. The SFC of samples at 45°C emulsion at sampling points Y (●) and Z (○) were 13.9 and 14.1%, and the product temperatures were 18 and 22.9°C, respectively. The SFC of samples at 50°C emulsion at sampling points Y (▲) and Z (△) were 15.6 and 15.8%, and the product temperatures were 17.7 and 22.9°C, respectively.

profile of the bulk oil could not be used directly to determine the SFC developed in the tube cooler during production. This was mainly due to the presence of other ingredients in margarine and the difference in crystallization conditions (tempering at 0°C) (1).

As the emulsion at its holding temperatures of either 40, 45, or 50°C was discharged into the tube cooler with constant cooling surface temperature, the amount of SFC developed was 15.9, 13.9, and 15.6%, respectively (Table 2). According to Yap *et al.* (21), the difference in the amount of SFC developed was due to the rate of crystallization of fats, which was higher during cooling than at isothermal temperature. However, the SFC developed after the B-unit (15.7, 14.1, and 15.8%, respectively) was slightly higher than the SFC in the tube cooler. This indicated that the pin worker had generated further crystallization (latent heat) as well as created mechanical heat, and melted some metastable crystals.

As the emulsion temperature was lowered, it was closer to the crystallization temperature with the same supercooling temperature (21). At a holding temperature of 50°C, the temperature difference between the holding and crystallization temperatures of bulk palm oil (22°C) was high. Thus, the onset of crystallization occurred toward the end of the tube cooler and the SFC was low. Crystallization then proceeded in the pin worker unit with an increase in temperature and some destruction of the crystal agglomerates. According to Andersen and Williams (7), the mechanical action of the pin

TABLE 3
SFC (%) of Palm Oil Margarine (28°C) Processed at Different Emulsion Temperatures

Emulsion temperature (°C)	Storage time at 28°C[a,b]				
	Day 1	Week 1	Week 2	Week 3	Week 4
40.0	$11.0 \pm 0.0^{a,A}$	$10.3 \pm 0.8^{b,B}$	$9.3 \pm 0.2^{c,B}$	$9.6 \pm 0.4^{e,B}$	$9.2 \pm 0.1^{f,A}$
45.0	$11.1 \pm 0.1^{a,C}$	$10.4 \pm 0.5^{b,D}$	$9.9 \pm 0.2^{d,D}$	$9.9 \pm 0.4^{e,D}$	$10.0 \pm 0.4^{g,C}$
50.0	$10.7 \pm 0.5^{a,E}$	$10.2 \pm 0.6^{b,F}$	$9.4 \pm 0.5^{c,d,F}$	$9.5 \pm 0.2^{e,F}$	$9.4 \pm 0.5^{f,g,F}$

[a]Means of 10 determinations ± SD obtained from two determinations daily for 5 d/wk.
[b]Means within columns with different letters (a–g) are significantly different ($P < 0.05$). Means within rows with different letters (A–F) are significantly different ($P < 0.05$). See Table 2 for abbreviation.

worker creates an extremely large number of small crystals and causes an additional increase in SFC.

At a holding temperature of 45°C, crystallization occurred earlier (shorter induction period) in the tube cooler. Metastable crystals (α-form) formed in the early stages of the crystallization process were transformed into a more stable crystal form with release of the heat of crystallization (1). At the same time, the heat released melted some of the crystals and reduced the SFC. Crystallization proceeded in the pin worker unit from the low SFC partially crystallized emulsion. The mechanical action of the pin rotor caused breakage of agglomerates so that the crystals were more dispersed. The SFC of the sample increased, but slowly.

At a holding temperature of 40°C, crystallization occurred more rapidly as seen by the higher SFC with release of latent heat. The difference between the holding temperature (40°C) and the crystallization temperature of palm oil (22°C) was 18°C, which was very close to the supercooling temperature (16.6°C). This suggested that crystallization might have taken place with a much shorter induction period (1). Mechanical action of the pin worker broke some of the already-formed crystal agglomerates in the tube cooler. Crystallization might have been completed (reached equilibrium) in the tube cooler, leaving very little crystallization to occur in the pin worker. This led to the lower SFC.

As shown above, the holding temperature of the emulsion had some influence on the product during processing. However, no statistically significant effect was observed on the physical properties of the margarine during storage at 28°C. Only slight differences were observed in SFC, consistency, and polymorphic behavior at several points during storage.

Table 3 shows that the SFC of the margarine was unstable during the first week of storage but stabilized after prolonged storage. Samples from 40 and 50°C holding temperatures had low SFC throughout the storage, despite their high SFC during processing. According to De Man *et al.* (22), this could be due to the melting of some crystals at the high storage temperature. The melted fat in the margarine provided opportunity for the crystals to reorganize, recrystallize, and eventually come to equilibrium. The sample at a holding temperature of 45°C had the lowest SFC during processing but maintained the highest SFC during storage. The storage temperature did not significantly affect the SFC of the product. The conditions employed during cooling and working had thoroughly distributed the crystals and formed a strong crystal network. High storage temperature did not lead to a substantial decrease in SFC during storage.

The consistency or penetration yield value (g/cm^2) of the experimental samples started high, declined in the first week, and eventually remained stable after the second week (Table 4). Faur (1) reported the same trend, but only for 5 d of storage. Moziar *et al.* (17) found that the hardness of shortening had no direct correlation with the SFC of the product.

Margarine produced at an emulsion holding temperature of 40°C had the hardest texture during storage, although this result seems contrary to the SFC (Table 3). However, hardness was correlated to the SFC in the tube cooler (point Y). As discussed earlier, an emulsion temperature of 40°C caused crystallization earlier in the tube cooler followed by less crystallization in the pin worker and the lowest product temperature. The crystal mixture formed in the tube cooler was worked rapidly in the pin worker, which led to the breakage of crystal agglomerates. The slow crystallization in the pin worker (lowest temperature) may have subsequently enhanced the formation of crystal aggregates and led to the formation of big crys-

TABLE 4
Penetration Yield Value (consistency: g/cm^2) of Palm Oil Margarine Processed Under Different Emulsion Temperatures

Emulsion temperature (°C)	Storage time at 28°C[a,b]				
	Day 1	Week 1	Week 2	Week 3	Week 4
40.0	$157 \pm 0.4^{a,A}$	$151 \pm 11^{b,A}$	$136 \pm 28^{d,A}$	$140 \pm 22^{e,A}$	$112 \pm 13^{g,B}$
45.0	$148 \pm 1.0^{a,C}$	$134 \pm 13^{c,D}$	$128 \pm 8^{d,D}$	$132 \pm 16^{e,D}$	$129 \pm 28^{g,D}$
50.0	$156 \pm 1.9^{a,E}$	$131 \pm 23^{c,F}$	$130 \pm 14^{d,F}$	$117 \pm 15^{f,F}$	$115 \pm 20^{g,F}$

[a]Means of 30 determinations ± SD obtained from six determinations daily for 5 d/wk.
[b]Means within column with different letters (a–g) are significantly different ($P < 0.15$). Means within rows with different letters (A–F) are significantly different ($P < 0.15$).

TABLE 5
Storage Study on the Polymorphic Transformation (28°C) of Palm Oil Margarine Processed Under Different Emulsion Temperatures[a]

Emulsion temperature (°C)	Spacings (°A)	Storage time at 28°C					
		Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
40	Long						
	13.96	W	W	W	W	W	W
	Short						
	4.57			VW	VW	VW	VW
	4.50			VW	VW	VW	VW
	4.31	S	S	S	S	S	S
	4.16	S	S	S	S	S	S
	3.99	VW	VW	VW	VW	VW	VW
	3.81	S	S	S	S	S	S
	Polymorph	β′	β′	β′ + β (β′ >>> β)	β′ + β (β′ >>> β)	β′ + β (β′ >>> β)	β′ + β (β′ >>> β)
45	Long						
	13.96	W	W	W	W	W	W
	Short						
	4.31	S	S	S	S	S	S
	4.16	S	S	S	S	S	S
	3.99	VW	VW	VW	VW	VW	VW
	3.81	S	S	S	S	S	S
	Polymorph	β′	β′	β′	β′	β′	β′
50	Long						
	13.96	W	W	W	W	W	W
	Short						
	4.31	S	S	S	S	S	S
	4.16	S	S	S	S	S	S
	3.99	VW	VW	VW	VW	VW	VW
	3.81	S	S	S	S	S	S
	Polymorph	β′	β′	β′	β′	β′	β′

[a]Abbreviations: S, strong; M, medium; W, weak; VW, very weak.

tals. There was also a transformation from β′ to β crystal at the end of the third week (Table 5).

The margarine sample produced when the emulsion holding temperature was 45°C had a lower penetration yield value than the sample produced when the emulsion was held at 40°C. The product had the lowest SFC at the tube cooler and the pin worker. However, the product had the highest SFC during storage. The polymorphic form of this product was stable in the β′ form through the fourth week of storage. It is likely that the crystals were more dispersed in this product and formed a good crystal network, which led to stable product consistency during storage.

The product made with emulsion at a holding temperature of 50°C had a high penetration yield value at week one, but this gradually declined toward the end of the fourth week. It may be hypothesized that the amount of crystal nuclei and crystal developed during processing was sufficient for the development of stable β′ crystals that lasted for more than 4 wk. The product softened during storage (at weeks 3 and 4), perhaps due to the reorientation of crystals into a more stable state. This process, however, did not involve significant polymorphic change and hardening (22).

In general, the experimental margarines were smooth and shiny with fat crystals mostly in their desired β′ polymorphic form during storage. The exception was the sample made from emulsion at 40°C holding temperature. This sample remained for only 2 wk in the β′ form, after which a mixture of β′ and β crystals was observed with more β crystal. The β crystal polymorph is the desired crystal form in margarine to have a smooth consistency and spreadability (8,19). According to De Man (19), the β′ crystal polymorph of shortening and margarine is on the order of 3–5 μm, whereas the β crystal form is larger than 50 μm. According to Chawla *et al.* (23), the β′ crystal polymorph occurs in single, needle-shaped crystals about 5–7 μm in length, whereas the β crystal polymorph is about 20–30 μm in size. The smaller the crystal size, the smoother the product, so the bigger crystals make the product appear coarse, grainy, and brittle (24).

Table 6 shows the softening points of the margarine samples. They ranged from 38.7 to 41.2°C, which was slightly higher than the dropping point of the original oil (38.6°C) during the first week of storage. The softening point of the sample made with 40°C holding temperature was in the range of 39.2–40°C, the sample with 45°C emulsion holding temperature varied from 38.8 to 41.2°C, and the sample with 50°C emulsion holding temperature varied from 39.7 to 39.2°C. Postmus *et al.* (25) showed that an increase of 2.2–3.3°C in the softening point of margarine above the dropping point of the animal fat used in the product indicated that a margarine with β crystal form had been produced.

TABLE 6
Softening Point (°C) of Palm Oil Margarine Processed Under Different Emulsion Temperatures

Emulsion temperature (°C)	Storage time at 28°C[a,b]			
	Day 1	Week 1	Week 2.	Week 3
40	$38.5 \pm 0.28^{a,A}$	$39.2 \pm 0.62^{b,A}$	$39.9 \pm 0.72^{c,A}$	$39.7 \pm 0.50^{d,A}$
45	$38.3 \pm 0.14^{a,B}$	$38.8 \pm 0.8^{b,B}$	$39.9 \pm 1.0^{c,B}$	$40.5 \pm 1.2^{d,B}$
50	$39.6 \pm 0.14^{a,C}$	$39.5 \pm 0.6^{b,C}$	$38.9 \pm 0.6^{c,C}$	$39.1 \pm 0.1^{d,C}$

[a]Means of six determinations ± SD obtained from two determinations daily for 3 d/wk.
[b]Means within columns with different letters (a–d) are significantly different ($P < 0.05$). Means within rows with different letters (A–C) are significantly different ($P < 0.05$).

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