Properties of Palm Oil Margarine During Storage: Effects of Processing Conditions

Miskandar Mat Sahri* and Nor Aini Idris*

INTRODUCTION

Margarine is a water-in-oil emulsion. The aqueous phase consists of water, salt and preservatives (Faur, 1996). The fatty phase, which contributes to the polymorphic behaviour of the margarine, is a blend of oils and fats (Nor Aini, 1992). Lecithin, distilled monoglycerides and diglycerides are common emulsifiers added together to the fatty phase with flavouring, colouring agents and antioxidants.

A good margarine should not be prone to oil separation, discolouration, hardening, sandiness, graininess and water separation (Chateris and Keogh, 1991). The oils and fats, process conditions and handling methods should be selected so as not to produce a strong crystal network (Haighton, 1965), crystal migration and transformation of β'- to β-crystals. In order to be smooth, creamy and homogenous, it is desirable for margarinee to be crystallized in the β-form. The β-crystal form, on the other hand, results in one that is post-hardened, brittle, grainy, sandy, oiled out and greasy (deMan *et al.*, 1989).

As proper solidification of oils and fats in margarine processing requires the right processing technique, the main concern, therefore, is the crystallization process. Processing parameters, such as emulsion temperature, agitation, flow rate of emulsion, cooling temperature and working are critical (deMan *et al.*, 1989). This study investigated the effects of processing conditions on the crystallization and physical properties of a palm oil margarine model during storage.

* Malaysian Palm Oil Board,
P .O. Box 10620,
50720 Kuala Lumpur,
Malaysia.
E-mail: miskand@mpob.gov.my

MATERIALS AND METHODS

Materials

The fat used in the study was refined, bleached and deodorized (RBD) palm oil 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 an emulsifier [distilled monoglycerides 90% monoester, SMP 69°C, source: fully hydrogenated palm oil from Danisco Ingredients (M) Sdn Bhd, Prai Industrial Estate, Pulau Pinang, Malaysia], water (filtered municipal supply) and vacuum-dried salt.

Chemical and Physical Analysis

The SMP and solid fat content (SFC) were determined according to MPOB Test Methods (2004).

Production of Margarine

A model of 80% RBD palm oil, 0.2% emulsifier, 16% water and 2.5% salt was used for the margarine. Palm oil was melted in a Memmert drying oven (854 UL 80, Schwabach, Germany) at 65°C then weighed into 50 kg production batches and a batch was placed in the mixing tank. The emulsifier was added to the palm oil in the ratio of 1:4. The water phase at room temperature (28°C) was then added slowly to the oil phase while agitating to form a good emulsion. Three batches were made with different emulsion temperatures – 40°C, 45°C and 50°C – and held for 10 min in the mixing tank prior to processing in a perfector pilot plant (Gerstenberg and Agger, Copenhagen, Denmark) at the Malaysian Palm Oil Board (MPOB). An intermediate crystallizer (C-unit) with a volume of 750 ml was attached in series to the tube cooler (A-unit) with a volume of 900 ml and scraped cooling surface of 0.063 m^2 area. The tube cooler for different runs was set at different temperature of 15°C, 20°C and 25°C respectively. The pin worker (B-unit) had a volume of 3 litre. The emulsion was pumped into the A-unit (at different throughput rates of 15, 30 and 45 kg hr^{-1}) where it was rapidly cooled. The scraper blades and the intermediate crystallizer were rotated at 500 rpm for all the three cases, whereas different pin worker speeds were used at 100, 200 and 300 rpm were used respectively.

Sampling Procedure

Samples were taken at three places to determine the development of SFC in the emulsions; from the mixing tank (sample X), exit of the scraper unit with the intermediate crystallizer (sample Y), and exit of the pin worker unit (sample Z) *(Figure 1)*. Duplicate samples were filled into NMR tubes, 0.80 cm in diameter and 2 cm height, and the SFC determined immediately (Miskandar *et al.*, 2002a). The time between sampling and reading was standardized at 50-60 s to minimize errors. The margarine made was collected in 400 ml tubs at the end of the processing line after the pin worker and stored at 28°C for evaluation.

Quality Assessment

The samples for analysis were placed in a 28°C incubator for 28 days. The consistency was determined by the penetration yield (g cm^{-2}) (Haighton, 1965; deMan *et al.*, 1989) using a cone penetrometer (Stanhope-Seta, Surrey, England) with a 40° cone, of weight 79.03 g with a penetration time of 5 s. The calculation was according to Haighton (1965), $KW/P^{1.6}$, where K=5840, W=79.03+ added weight, and P= mean of penetration readings. Six readings were taken from each sample every day, with different sub-samples.

The isothermal SFC (Miskandar *et al.*, 2002a) was determined daily, five days a week. A solid margarine sample (from the same tub previously used for penetration) was loaded into an NMR tube of 0.8 cm diameter to a height of 2 cm (in duplicates), using a stainless steel piston. The sample was loaded very carefully to prevent trapping any air space in it. The sample in adiabatic condition was measured by pulse NMR. Polymorphic changes (Miskandar *et al.*, 2002b) were determined every five days 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

Data analysis was performed by ANOVA using Microsoft Excel 2000.

Figure 1. Margarine pilot plant.

RESULTS AND DISCUSSION

Effect of Emulsion Temperature

The two most prominent changes occurring during processing of margarine are the increase in the SFC and temperature of the product (Miskandar *et al.*, 2002a). According to Faur (1996) and Haighton (1976), these changes are related to the crystal development. The increase in SFC releases the latent heat of crystallization that raises the product temperature. Thus, when the processing conditions are held constant, while varying the mixing temperature at Point X as shown in *Figure 1*, the temperatures at Points Y and Z will vary as well.

As the emulsions at their holding temperatures of 40°C, 45°C and 50°C were discharged into the tube cooler at constant surface temperature, the SFCs produced were 15.9%, 13.9% and 15.6%, respectively. After the pin-worker, the SFC averaged 15.7%, 14.1% and 15.8%, respectively. Thus, at the emulsion temperatures of 45°C and 50°C, the SFCs after the pin-worker were only slightly higher than after the tube cooler, suggesting that the pin-worker, while promoting crystallization, generated mechanical heat as well which, in addition to the latent heat released, melted some of the metastable crystals. The mechanical action of the pin-worker also destroyed some of the crystal bonds creating an extremely large number of small crystals and increasing the SFC (Andersen and Williams, 1965).

The consistency, or penetration yield (g cm^{-2}), of the samples started off high but declined in the first week and stabilized after the second *(Figure 2)*. The margarine produced from the emulsion at holding temperature 40°C exhibited the highest consistency at week 1 but was in an unstable state after further storage, while the other two samples took only one week to reach equilibrium. Crystallization occurred earlier in the tube cooler resulting in less crystallization in the pin-worker as indicated by the low product temperature. The crystallization in the tube cooler was not very homogenous, and thus incomplete, but the agitation by the pin-worker broke up some of the crystal agglomerates. The little crystallization (as evidence by the low product temperature), and broken crystal bonds enhanced the formation of crystal aggregates that eventually led to the formation of bigger crystals as shown by the transformation of β'- to β-crystals at the end of the fourth week (*Table 1*).

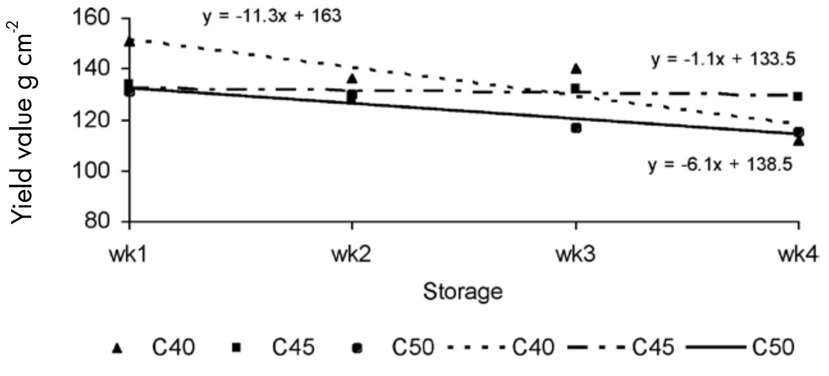

Figure 2. Penetration yields (consistency) (g cm^{-2}) of palm oil margarine processed from emulsions at different temperatures.

Effect of Emulsion Flow Rate

The speed at which the margarine emulsion passes the scrape-surface tube cooler and pin-worker system will affect the end product: too slow, the margarine becomes hard and brittle as the emulsion is cooled too rapidly; too fast, the crystals attach to each other instead of orientating themselves in a better position (Timms, 1994). On the other hand, too high a feeding rate gives insufficient cooling, promoting post-crystallization and hardening, especially in packet margarines.

The consistencies (penetration yields) of the margarine samples processed at different flow rates are shown in *Figure 3*. The margarines from flow rates of 15 kg hr^{-1} and 30 kg hr^{-1} were spreadable on bread but that from the 45 kg hr^{-1} flow rate showed some greasiness although without oil separation. The initial consistencies of all the samples were almost the same but increased at different rates during storage. With storage they became more different in their consistencies, especially after two weeks, owing to the crystal arrangement and degree of crystallization. The consistent trends during storage for all the samples were very similar to that for industrial margarine (Haighton, 1965).

The consistency of the sample from flow rate 15 kg hr^{-1} increased sharply from the first week until the third week. This sample started with a high SFC at filling, and thus was expected to have high consistency in the first week of storage. The high SFC also indicated the formation of a strong crystal network with narrow capillaries in between the crystals. This condition does not allow easy crystal movement and therefore caused the margarine to

TABLE 1. POLYMORPHIC TRANSFORMATION OF PALM OIL MARGARINES MADE FROM EMULSIONS AT DIFFERENT TEMPERATURES DURING STORAGE AT 28°C

Emulsion temp (°C)	Storage time at 28°C			
	Wk 1	Wk 2	Wk 3	Wk 4
40	β′	β′	β′+β (β′>>>β)	β′+β (β′>>>β)
45	β′	β′	β′	β′
50	β′	β′	β′	β′

firm up until the third week (Manley, 1983). Only when some of the low melting glycerides started to melt during storage did the consistency start to decrease.

The margarines were mixtures of β- and β′-crystals, with more β′ in the first two weeks (*Table 2*). This shows palm oil as a β′-crystal promoter (Hui, 1996), and able to retain its polymorphic behaviour at different emulsion flow rates. The results also show that the process condition can delay the transformation of β′- to β form, as found by Yap *et al.* (1989) from blending palm oil with several other vegetable oils.

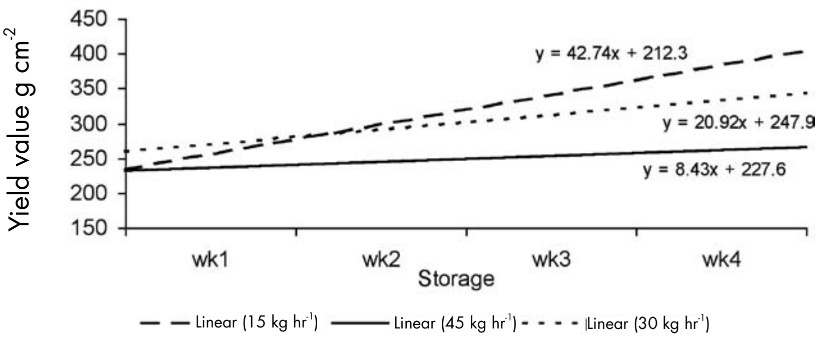

Figure 3. Penetration yields (g cm^{-2}) for stored samples of palm oil margarines made at different flow rates.

Effect of Scraped-Surface Tube Cooler Temperature

When a bulk oil crystallizes, the whole mass does not do so at the same time. Instead, the process starts at discrete sites where the temperature has fallen sufficiently low for crystallization points, or nuclei, to form (Miskandar *et al.*, 2002b). Thus, when a molten fat cools naturally, a granular product is produced due to the slow crystallization of the individual glycerides (Lawler and Dimick, 1989). With faster cooling, the crystals become smaller and more uniform (Che Man and Swe, 1995). If instantaneous chilling occurs, the high and low melting TAGs will develop mixed crystals (deMan *et al.*, 1989). Further, by stirring, the high and low melting TAGs crystallize at the same time causing rapid cooling (Borwanker *et al.*, 1992). Thus, the 15°C emulsion passing through received the greatest of cooling and formed the most nuclei and crystals, followed by the sample at 20°C and, finally, the sample at 25°C. The margarine samples of 15°C, 20°C and 25°C contained 23.5%, 17.7% and 13.2% SFCs, respectively, at Point Y.

As the 15°C emulsion passed out of the tube cooler, crystallization continued rapidly in the pin-rotor but with little net crystal growth. The mechanical activity of the pin-worker destroyed some of the crystal structure, thus reducing the

TABLE 2. POLYMORPHIC TRANSFORMATION DURING STORAGE FOR PALM OIL MARGARINE PROCESSED AT DIFFERENT FLOW RATES

Flow rate (kg hr^{-1})	Storage time at 28°C			
	Wk 1	Wk 2	Wk 3	Wk 4
15	β′+β (β′ >>β)	β′+β (β′ >>β)	β′+β (β =β′)	β′+β (β =β′)
30	β′+β (β′ >>β)	β′+β (β′ >>β)	β′+β (β = β′)	β′+β (β = β′)
45	β′+β (β′ >>β)	β′+β (β′ >>β)	β′+β (β=β′)	β′+β (β=β′)

SFC (Miskandar *et al*., 2002b). Crystal development in the sample at 20°C was less rapid in the tube cooler, but there was higher net development in the pin-worker. The least crystal development in the tube cooler was in the sample at 25°C. However, agitation in the pin-worker promoted its crystallization the most.

Destruction of the crystal agglomerates reduced the crystal size and caused a more even crystal dispersion. The penetration yields for the samples of 15°C and 20°C were low, but gradually increased with storage, as was also the case with the sample of 25°C, producing a more consistent product (*Figure 4*).

The crystal polymorphic development of the margarines during storage is shown in *Table 3*. The samples had mixtures of β'- and β-crystals with more β' in the sample of 15°C, more β in the sample of 20°C and predominantly β-crystals in the sample of 25°C at the start of storage.

Effect of Pin-Worker Speed

The pin-worker, besides further crystallizing the emulsion, also physically breaks up and works the crystals to improve the texture of the final product. Heertje (1993) reported that high pin-worker speed gives a soft and overworked product in spreads. The mechanical work also raises the temperature of the margarine in the pin-worker by 2°C or more by the heat generated, and the latent heat of crystallization released (Hui, 1996). This study found a temperature increase of 4.8°C -6.1°C after passage through the pin-worker. The increased temperature at Point Z was mainly due to the release of more heat of crystallization rather than that due to agitation and working of the emulsion (Hui, 1996).

As the emulsion passed through the tube cooler, it was super-cooled by the refrigerated tube surface to initiate crystallization. The difference in SFC between the two speeds of 100 rpm and the 300 rpm was significant, suggesting that an increase in retention time is required at the higher speed, similar to what was observed by Hui (1996). With constant emulsion flow rate, faster rotation of the pin-worker created more resistance to the product flow, thus slowing it down. The rotating blade of the scraped-surface heat exchanger then forced more emulsion onto the refrigerated surface resulting in more heat extracted. The emulsion therefore cooled more, reducing the induction time needed. The high melting glycerides started to solidify in the tube cooler and increased the SFC. The pre-crystallized emulsion was then passed to the pin-worker where further crystallization and homogenization occurred.

The subsequent gradual increase in consistency of the sample from 100 rpm could have been due to insufficient agitation of its crystal. This led to a stronger crystal network being developed during storage. The consistency of the sample from 200 rpm, however, only increased until the third week. The final product was low in consistency, but did not suffer any oil separation. The sample from 300 rpm was subjected to greater agitation and crystal agglomerate destruction, resulting in a large number of very small crystals. According to Haighton (1976), very small aggregates can form a very compact crystal structure that gives rise to a hard product with heavy mouth feel. The experimental margarines were smooth and free from oil separation, complying with the criteria for good quality margarine; however, they were not in the desired β'-crystal polymorph. *Table 4* shows that the pin-worker speeds used did not delay the reversion of β'- to β-crystals for longer than two weeks.

As palm oil is the main contributor of β'-crystals in margarine, the observation in *Table 4* suggests that although palm oil has the greatest tendency to be in β'-crystal form, the appropriate processing and storage conditions can also provide the environment for the transition from β' to β.

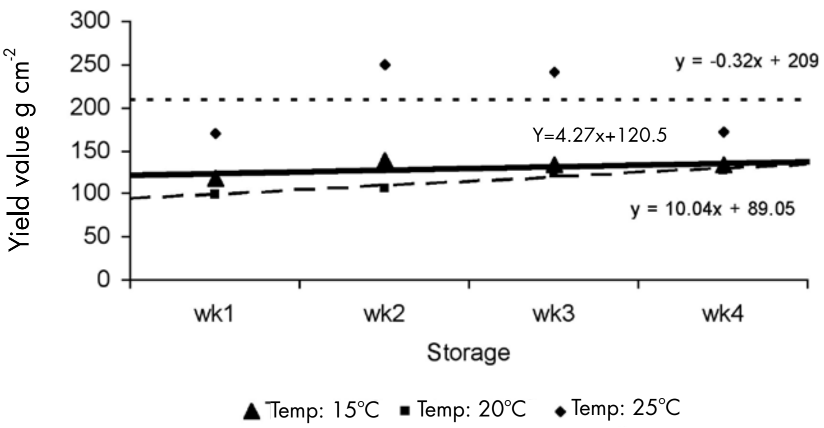

Figure 4. Penetration yields (consistency) (g cm^{-2}) of palm oil margarines processed at three tube cooler temperatures and stored at 28°C.

TABLE 3. POLYMORPHIC TRANSFORMATION OF PALM OIL MARGARINES PROCESSED AT DIFFERENT TUBE COOLER TEMPERATURES AND STORED AT 28°C

Tube cooler temp. (°C)	Storage time		
	Wk 2	Wk 3	Wk 4
15	β+β′ (β′ >>β)	β+β′ (β′ >β)	β+β′ (β = β′)
20	β+β′ (β′ >β)	β+β′ (β = β′)	β+β′ (β > β′)
25	β+β′ (β >>β′)	β+β′ (β >>>β′)	β+β′ (β >>>β′)

TABLE 4. POLYMORPHIC TRANSFORMATION OF PALM OIL MARGARINES PROCESSED AT DIFFERENT PIN-WORKER SPEEDS

Pin-worker speed (rpm)	Intensity of changes in polymorphs stored at 28°C over time			
	Wk 1	Wk 2	Wk 3	Wk 4
100	β′+β (β=β′)	β′+β (β=β′)	β′+β (β>β′)	β′+β (β>β′)
200	β′+β (β=β′)	β′+β (β=β′)	β′+β (β>β′)	β′+β (β>β′)
300	β′+β (β=β′)	β′+β (β=β′)	β′+β (β>β′)	β′+β (β>β′)

CONCLUSION

Reasonably good model margarine (after storage) can be produced by having correct process conditions, such as maintaining an emulsion temperature of about 10°C above its SMP, flow-rate of 45 kg hr^{-1}, super-cooling temperature of about 20°C and pin-worker speed of 200 rpm. It was also observed that correct process conditions can also delay the transformation of β′- to β crystal form during storage.

ACKNOWLEDGEMENTS

The authors would like to thank the Malaysian Palm Oil Board for permission to use the perfector pilot plant at the Oils and Fats Technology Centre (OFTEC). Thanks are also due to the staff of OFTEC for their technical assistance and facilities used.

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