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Rheological, chemical and DSC thermal characteristics of different types of palm oil/palm stearin-based shortenings

[1,*]Abdul Azis, A., [1]Mohamud, Y., [1]Roselina, K., [2]Boo, H. C., [3]Nyuk, L. C. and [1]Che Man, Y. B.

[1]*Department of Food Technology, Faculty of Food Science and Technology, Universiti Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia.*
[2]*Department of Food Service and Management, Faculty of Food Science and Technology, Universiti Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia.*
[3]*Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia*

Abstract: This study was carried out to evaluate the physical and chemical properties of different types of shortenings, formulated by mixing refined, bleached, and deodorized palm oil and palm stearin (PO:PS) in the following ratios: 100:0, 80:20, 60:40, 50:50, 40:60 and 20:80 and 0:100. The properties of experimental and commercial shortenings were investigated using four different analytical techniques, namely high performance liquid chromatography (HPLC), gas chromatography (GC), differential scanning calorimetry (DSC) and controlled stress rheometer. In addition, iodine value (IV) analysis was carried out. The results revealed that the prominent fatty acids in the products were palmitic (44.88-61.91%), oleic (26.24-39.14%) and linoleic (6.13-11.68%). At the same time, triacyglycerols (TAG), such as OOO, OOP and OOS, were found to decrease, while PPO increased due to the increase in the palm stearin content of the shortenings. Higher viscosity and more storage (G′) or loss (G″) modulus properties were noted in the experimental and commercial shortenings containing higher and lower concentrations of palm stearin and palm oil, respectively. Certain parameters such as the onset, peak and endset temperatures (ºC) were detected for both the melting and cooling data. However, increasing the palm stearin concentrations in the samples was shown to have caused increases in the endset temperature and peak height, and vice versa. Thus, chemical and physical properties of the formulated shortenings may influence the quality of baked products.

Keywords: shortening, DSC thermal properties, rheology, palm oil, palm stearin, chemical properties

Introduction

Palm oil has become one of the leading vegetable oil for edible applications because of its good quality and properties. Approximately 90% of palm oil is used in food products (Idris et al., 1989; Ong et al., 1995). Palm oil (PO) can be divided into palm stearin (solid fraction) and palm olein (liquid fraction). Meanwhile, refined, bleached and deodorized palm oil (RBDPO) is an important oil source for the food industry because it has good crystallization properties, whereby it tends to crystallize as β'. It is important to note that RBDPO is normally combined with other fat and/or oil, among other, to produce a product that melts quickly on the palate (Mat Dian et al., 2006).

The use of PO can be maximized by employing modification processes such as fractionation, blending, interesterification and hydrogenation. However, blending and interesterification are the most commonly known practical processes applied in the production of shortenings. Shortening is a product that is made completely from oil and fat, and it can be either straight or in mixture of the two for the preparation of food items (Osman and Aini, 1999). Blending is considered as the simplest modification process for fat and oil. Although requirements for fat products have become more sophisticated, there are correspondingly fewer ways in which appropriate specifications can be met, apart from simply blending natural fat and oil (Mat Dian et al., 2006). Being a less expensive product, palm stearin (PS) is very economical for shortening formulation, and it helps improve plasticity of shortenings (Aini and Maimon 1996).

The level of saturated fatty acids in the triglyceride determines the hardness and plasticity of the shortening, and it plays an important role in the properties of the final product (Qarooni, 1996). In

*Corresponding author.
Email: *abdulazis@putra.upm.edu.my*
Tel: +603-89468354, Fax: +603-89423552

particular, differential scanning calorimetry (DSC) has been increasingly utilized for thermodynamic characterization of edible oil and fat (Tan and Che Man, 2002). DSC heating curves give valuable information about the melting and solidification properties of a fat. In addition, it can be used to determine crystallization curves whereby it can show at what temperature the crystals start to form (Lai et al., 2000).

On the other hand, rheology is mainly concerned with the relationship between strain, stress and time. When subjected to external forces, solid will deform, whereas liquid will flow (Liu et al., 2008). It is a well-known fact that fat is a viscoelastic material at small deformations. The properties of viscoelastic fat products can be categorized into elastic and viscous, while shear modulus (G) comprises of two components, namely storage modulus (G′) and loss modulus (G″). The G′ reflected the solid-like characteristic of fats, particularly the strength of the links between the clusters of fat crystals, whereas G″ denotes the liquid-like characteristic of fat (Tang and Marangoni, 2006). Viscosity measurements are used to monitor changes in shortening rheology due to crystallization. However, Bell et al. (2007) and Jirasubkunakorn et al. (2007) utilized a controlled stress rheometer to study fat rheology. Physical and chemical properties of PO as a semi-solid vegetable oil make it more suitable in many food product formulations including shortening and margarine (Wan Rosnani et al., 2007). Therefore, the aim of this study was to determine the DSC thermal properties, rheology and chemical characteristics of different types of RBD palm oil/palm stearin-based shortenings.

Materials and Methods

Materials

Samples of RBD palm oil and RBD palm stearin, with IV of 52.3 and 31.2 respectively, were purchased from a local refinery located in Selangor, Malaysia. Meanwhile, emulsifiers of diacetyl tartaric acid ester of mono-diglycerides (DATEM) and distilled monoglyceride (DMG), with IV of 40 and 105 respectively, were purchased from Danisco Malaysia Sdn. Bhd. a company which is based in Penang, Malaysia. All the chemicals and solvents used were of analytical or HPLC grade.

The preparation of shortenings

RBD palm oil and RBD palm stearin were blended in their respective proportions of 100:0, 80:20, 60:40, 50:50, 40:60, 20:80 and 0:100 (w/w). Each blend was added with 6.25% of DMG and 6.25% of DATEM. Shortenings were prepared after complete melting of the oils at 70ºC using magnetic stirring, and the shortenings obtained were then stored at room temperature (28ºC).

Fatty acid composition

The fatty acid compositions of the shortenings were analyzed after converting the fatty acids into corresponding fatty acid methyl esters (FAME). All the samples were melted in oven prior to use, and 50mg of the sample was weighed (Chu et al., 2002). After methylation, the composition of fatty acid was determined using Hewlett-Packard 6890 chromatography (GC) that was equipped with an auto injector and a flame-ionization detector (FID) using a fused silica capillary column (60.0m×320µm×0.25µm film thickness, id-BPX70) at 260°C maximum, with Helium as the carrier gas, at a flow rate of 1.6mL/min. The injector and detector temperatures were set at 220°C and 240°C, respectively. Meanwhile, the oven temperature was programmed in two stages, as follows: first from 50°C to 180°C (8°C /min), and then from 180ºC to 240ºC (8ºC /min) (Mamat et al., 2005). Each analysis was conducted in triplicate, with a run time of 35.6min.

Iodine value

The IVs of the samples were determined according to the procedure described in the AOCS method (AOCS, 1988). The sample (0.5g) was diluted in 20ml of cyclohexane (in this analysis, cyclohexane was used in place of chloroform) and 25ml of the Wijs solution (ICl) was added to halogenate the double bonds. After placing the bottles in the dark for 1hour, the mixtures were reacted with 20ml of potassium iodine and 100ml distilled water. Free I_2 was measured by titration with 24.9g/l $Na_2S_2O_3.5H_2O$ using starch (1.0g/100ml) as an indicator. IV was calculated as cg I_2 adsorbed/g sample. The iodine value of each sample was determined in triplicate.

Triacyglycerol (TAG) composition

The TAG composition was determined using a high-performance liquid chromatography system (JASCO PU-1580) that was equipped with a commercially packed RP-18 column (25cm×4.6mm; Merck, Darmstadt, Germany), injector, Jasco RI-1530 detector, and Borwin software. The mobile phase was a mixture of acetone-acetonitrile at a ratio of 70:30 (v/v), and at a flow rate of 1 mL/min (Mamat et al., 2005). The TAG peaks were identified by comparing the retention times of the TAG standards and those derived in the work of Chen et al. (2007), Marikkar et al. (2002), as well as Tan and Che Man (2002). Each sample was chromatographed three times and

the data were reported as percent areas.

Viscosity and viscoelastic measurements

A Haake RT 20 controlled-stress and rate rheometer (ROTOVISCO, Germany) was used to measure the viscosity and viscoelasticity behaviours of the shortening samples. Meanwhile, the oscillatory amplitude sweeps were carried out to determine the viscoelastic behaviour for each sample (Bell et al., 2007), and the CS/CR-Rotation Ramp method was used to investigate the viscosity. All the samples were carefully scooped out with a flat-based stainless steel to minimize sample deformation. Dynamic measurements (oscillation) were performed with a 35 mm diameter (Laia et al., 2000) with serrated parallel plate geometry (PP35Ti). The storage modulus (G′) and loss modulus (G″) were determined every 1.36 minute, and at a frequency of 1 Hz. The viscosity and viscoelastic measurements were taken at each step and each sample was tested in triplicate.

Differential scanning calorimetry (DSC)

Differential scanning calorimetry (DSC) measurements were carried out using the Mettler-Toledo DSC 823E1400 model. In these measurements, nitrogen was used as purge gas. The samples containing 6 mg were loaded to the middle of the aluminium pans using a small spatula and hermetically sealed. Indium (melting temperature 156.6ºC, ΔHf =28.45J/g) and n-dodecane (melting temperature -9.65ºC, ΔHf = 216.73 J/g) were used to calibrate the instrument and an empty covered pan was used as a reference. The following temperature programmes were used to perform the melting and cooling measurements on each sample: 70ºC isotherm for 5min, cooled to -60ºC and held for 5min. The same sample was then heated from -60ºC to 70ºC (Rosnani et al., 2007; Tan and Che Man, 2002). It is important to note that triplicate analyses were carried out per sample.

Statistical analysis

Both the means and standard deviations (SD) were calculated using the MINITAB (version 14.0, Minitab Inc.) statistical software. Meanwhile, the one-way analysis of variance (ANOVA), with the Tukey's multiple comparison at a family error rate of 0.05, was performed to test the significance.

Table 1. The compositions of fatty acid in the experimental shortenings of RBD palm oil/palm stearin blends and commercial shortenings

Shortenings Ratio of PO:PS	Fatty acid compositions (wt %)					Iodine value
	C14:0	C16:0	C18:0	C18:1	C18:2	
Control	1.02± 0.06[a]	44.88± 0.40[a]	4.07±0.05[a]	38.3± 0.60[a]	11.68± 0.3[a]	52.45±0.89[a]
80:20	1.25±0.08[b]	48.38±0.60[be]	3.54±0.20[a]	37.19±0.40[b]	9.64±0.20[bc]	48.7±0.36[b]
60:40	1.17±0.04[b]	51.17±1.20[b]	3.81±0.01[a]	34.81±1.00[c]	9.04±0.30[b]	44.7±0.30[c]
50:50	1.24±0.05[b]	53.24±0.30[c]	3.83±0.10[a]	33.18±0.20[d]	8.51±0.01[c]	43.6±0.36[cd]
40:60	1.25±0.03[ab]	53.62±0.80[c]	3.68±0.30[a]	32.88±0.80[d]	8.57±0.20[c]	42.1±0.20[d]
20:80	1.31±0.04[c]	58.62±0.60[d]	3.95±0.08[ab]	28.70±0.40[e]	7.42±0.10[d]	37.3±0.90[e]
0:100	1.19± 0.01[b]	61.91±1.06[cd]	4.52± 0.02[b]	26.24± 0.90[f]	6.13± 0.09[d]	31.22± 0.51[f]
Commercial	1.15±0.06[ab]	47.64±1.20[e]	3.86±0.20[a]	39.14±0.60[a]	8.21±0.40[bc]	49.8±0.00[g]

The data were obtained from the mean value of three replications.

[a] values are means ± SD; Means with same letter within each column were not significantly different ($p<0.05$).

Table 2. Triacylglycerol profile (% peak area) of different types of RBD palm oil/palm stearin-based shortenings

TAG	Types of shortenings (ratios of PO:PS)							
	Control	**80:20**	**60:40**	**50:50**	**40:60**	**20:80**	**0:100**	**Commercial**
MMM	0.53±0.0[a]	0.62±0.01[b]	0.65±0.05[c]	0.54±0.025[abc]	0.61±0.02[bc]	0.64±0.00[bc]	1.03±0.04[b]	0.43±0.03[d]
PLL	1.48±0.08[a]	2.28±0.03[ac]	1.84±0.45[bcd]	2.06±0.12[d]	2.05±0.019[de]	1.89±0.05[def]	1.70±0.15[ac]	2.48±0.01[b]
MPL	0.39±0.06[a]	0.27±0.02[a]	0.33±0.01[a]	0.32±0.056[a]	0.32±0.02[a]	0.23±0.09[a]	0.32±0.25[a]	0.66±0.03[b]
OOL	1.73±0.01[a]	1.76±0.05[a]	1.65±0.08[a]	1.54±0.03[b]	1.54±0.05[b]	1.53±0.03[b]	1.50±0.11[b]	1.68±0.056[ab]
PLO	11.0±0.37[a]	10.4±0.26[a]	9.89±0.53[ab]	9.21±0.11[b]	9.03±0.22[bc]	8.70±0.03[c]	7.39±0.46[d]	10.11±0.34[a]
PPL	8.04±0.21[a]	11.0±0.04[b]	9.37±1.68[a]	10.68±0.11[b]	10.89±0.12[b]	9.77±2.02[a]	10.7±0.41[b]	10.63±0.26[b]
OOO	3.86±0.03[a]	3.66±0.14[ab]	3.39±0.14[b]	3.19±0.08[b]	3.12±0.05[b]	3.01±0.17[b]	2.56±0.06[c]	3.70±0.10[a]
OOP	25.7±0.07[a]	22.9±0.45[b]	21.6±1.01[bc]	20.41±0.53[c]	19.80±0.28[c]	19.29±0.93[c]	16.5±0.70[d]	23.70±0.61[bc]
PPO	32.2±0.17[a]	32.4±0.74[ab]	34.21±1.20[ab]	34.31±0.34[b]	36.65±0.45[c]	39.8±1.75[cd]	40.8±0.98d	32.26±0.70[ab]
PPP	5.61±0.25[a]	5.79±1.50[ab]	7.57±1.17[ab]	8.42±1.16[b]	6.44±1.08[ab]	5.11±0.63[ab]	7.66±1.86[ab]	5.24±1.99[ab]
OOS	2.37±0.13[a]	1.95±0.08[b]	1.93±0.16[bc]	1.79±0.03[bc]	1.71±0.01[c]	1.60±0.04[c]	1.39±0.07[d]	2.22±0.07[ab]
POS	5.4±0.17[ab]	5.24±0.04[a]	5.50±0.29[a]	5.39±0.0[ac]	5.76±0.08[b]	6.43±0.21[d]	6.11±0.41[cd]	5.34±0.16[abc]
PPS	1.07±0.08[a]	1.18±0.26[ac]	1.55±0.19[b]	1.63±0.31[b]	1.55±0.16[b]	1.47±0.27[ab]	1.82±0.31[bc]	1.02±0.30[abc]
SOS	0.54±0.02[a]	0.50±0.03[a]	0.50±0.04[a]	0.51±0.01[a]	0.51±0.01[a]	0.50±0.05[a]	0.53±0.02[a]	0.52±0.02[a]

[a] values are means ± SD; Means with the same letter within each row are not significantly different (n= 3, $p<0.05$).
Abbreviations: TAG, triacylglycerol; M= myristic; L= linoleic; O= oleic; P= palmitic, and S= stearic.

Results and Discussion

Fatty acid composition

The fatty acid analysis of the samples showed that the sample containing 0% palm oil and 100% palm stearin had that highest proportion of palmitic acid, but with the lowest proportions of oleic acid and linoleic acid which are the dominant unsaturated fatty acids as compared to the other samples. In more specific, saturated fatty acids include three types of fatty acids, namely myristic (C14:0), palmitic (C16:0), and stearic (C18:0) acids, while unsaturated fatty acids include two types of fatty acids, namely oleic (C18:1) and linoleic (C18:2) acids. Other fatty acids were also present but in small amounts. The major fatty acids found in the shortening samples were palmitic acid and oleic acid. The detailed fatty acid compositions (mean ± SD) of all the samples are presented in Table 1.

Increasing the concentration of palm stearin and decreasing the content of palm oil in the blends have caused a gradual increase of the total of palmitic acid (44.88±0.4, 48.38±0.6, 51.17±1.2, 53.24±0.3, 53.62±0.8, 58.62±0.6, 61.91±1.06 and 47.64±1.2 for control, 80:20, 60:40, 50:50, 40:60, 20:80, 0:100 and commercial, respectively). Meanwhile, the total of oleic acid was found to gradually decrease with the increasing content of palm stearin and decreasing the concentration of palm oil (38.3±0.6, 37.19±0.4, 34.81±1.0, 33.18±0.2, 32.88±0.8, 28.70±0.4, 26.24±0.9 and 39.14±0.6 for control, 80:20, 60:40, 50:50, 40:60, 20:80, 0:100 and commercial, respectively).

The iodine value is a measure of the unsaturation of fat and oil. It is one of the most important parameters for measuring the quality of olein (Mamat et al., 2005). This study has shown that the iodine value of the experimental shortenings decreased significantly ($p<0.05$) with the increase of palm stearin content and vice versa (see Table 1).

Triacyglycerol (TAG) composition

The triacyglycerol (TAG) structure of fat is an important parameter in the development of food products, such as shortening, confectionary and margarine. They constitute the major components of TAG, but with small proportions of di- and mono-glycerides. In particular, TAGs appear as different crystal structures because they have long hydrocarbon chains that can be packed into different crystal lattices which cause polymorphism (Szyd owska-Czerniak et al., 2005).

Table 2 shows the TAG profile of different types of RBD palm oil/palm stearin shortenings, as well as commercial shortening. The identification of the TAG peaks of the shortenings is based on the TAG standards, as well as according to Chen et al. (2007), Marikkar et al. (2002), Tan and Che Man (2002). In total, fourteen TAGs were detected in the RBD palm oil, palm stearin-based shortenings and commercial

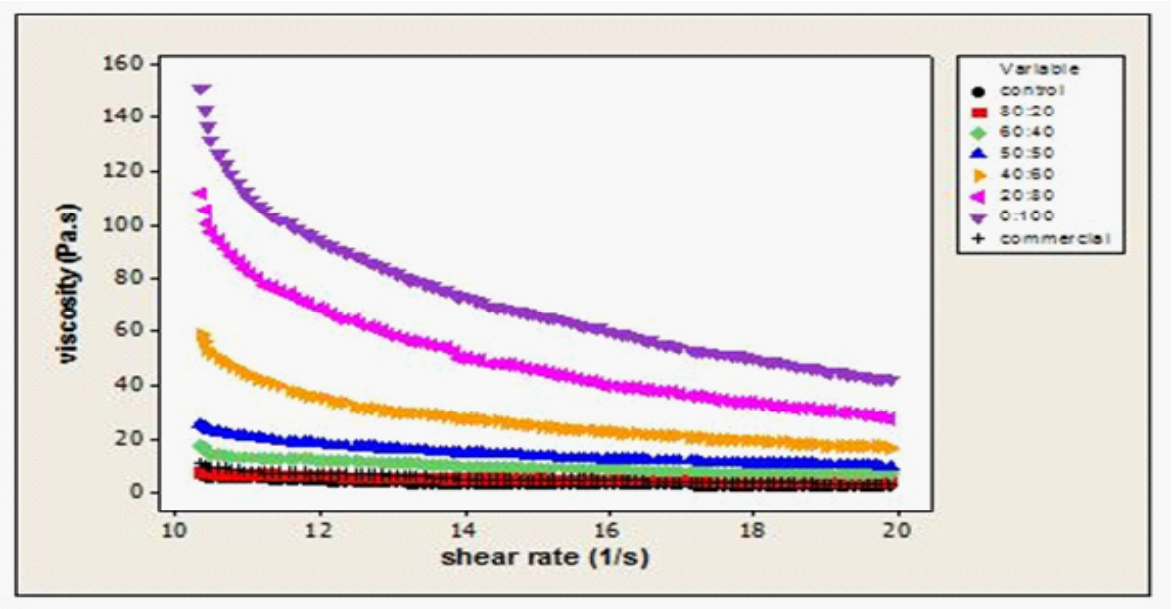

Figure 1. Variation of the viscosities (Pa.s) for the different types of RBD palm oil/ palm stearin and commercial shortenings, as a function of shear rate (1/s)

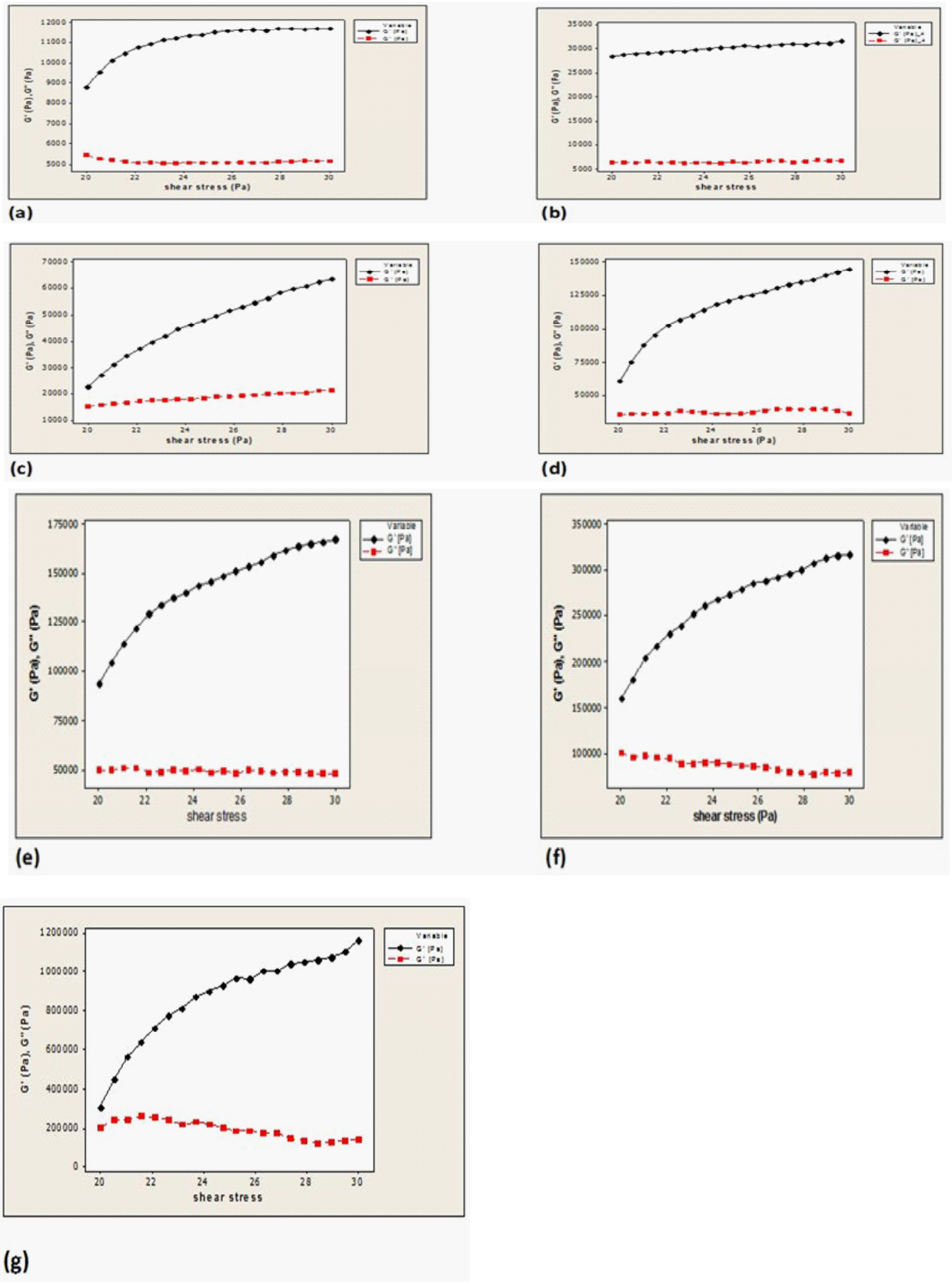

Figure 2. Changes in storage (G′) and loss (G″) modulus at 1Hz for the shortenings as a function of shear stress (Pa), (a) 80:20; (b) commercial; (c) 60:40; (d) 50:50; (e) 40:60; (f) 20:80; (g) 0:100.

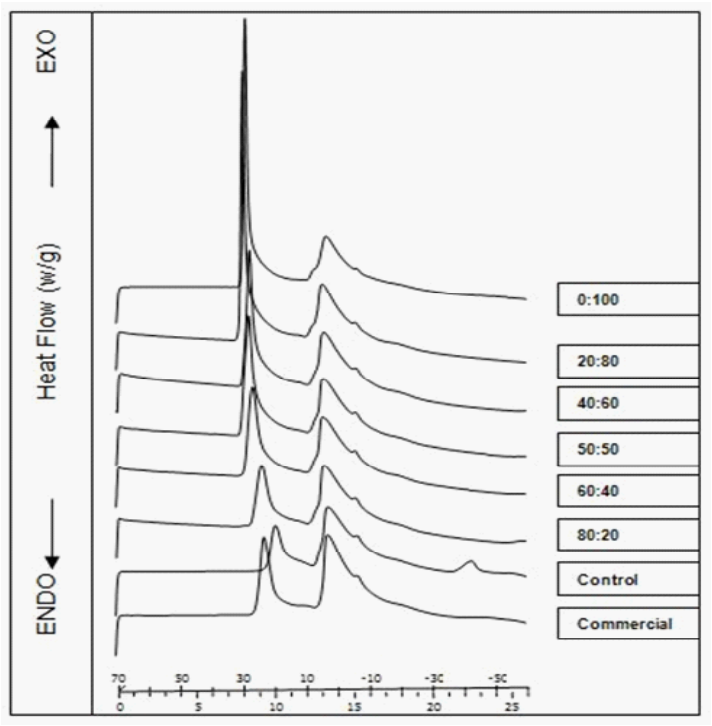

Figure 3. Differential scanning calorimetry cooling curves of different types of shortenings based on RBD palm oil/palm stearin blends and commercial shortening

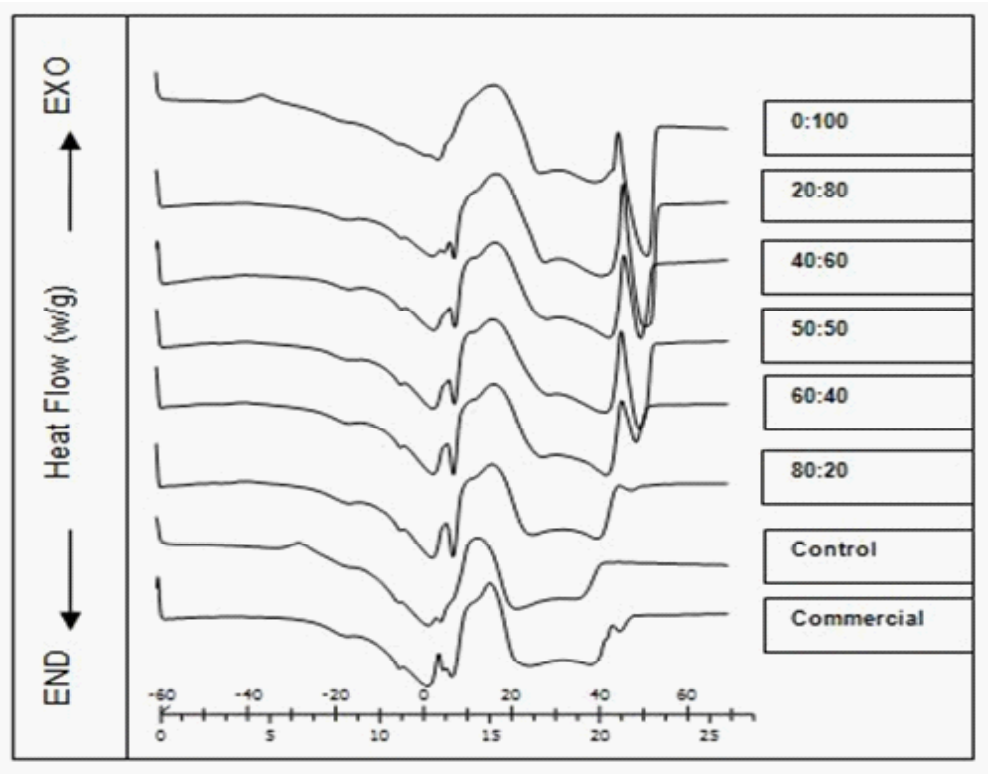

Figure 4. The differential scanning calorimetry heating curves of different types of shortenings based on the RBD palm oil/palm stearin blends and commercial shortening

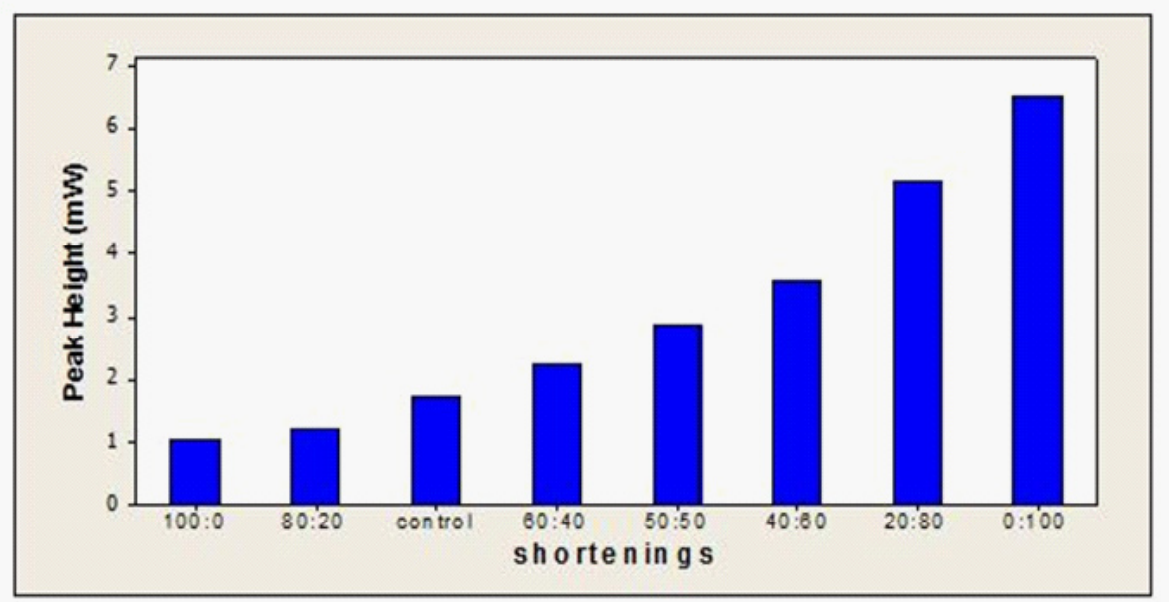

Figure 5. The peak heights (mW) of major exothermic event of the cooling thermograms for the shortenings

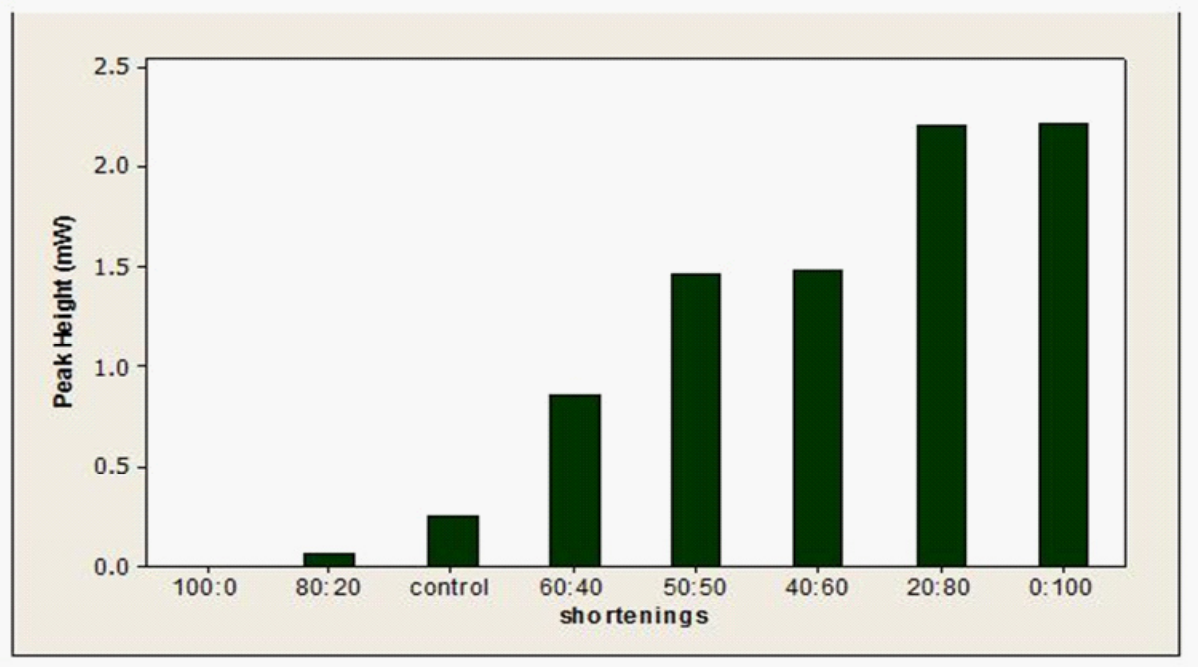

Figure 6. The peak heights (mW) of the major endothermic event of the heating thermograms for the shortenings

shortening, namely 1: MMM, 2: PLL, 3: MPL, 4: OOL, 5: PLO, 6: PPL, 7: OOO, 8: OOP, 9: PPO, 10: PPP, 11: OOS, 12: POS, 13: PPS, 14: SOS, whereby M, L, O, P and S represent myristic, linoleic, oleic, palmitic and stearic, respectively. Meanwhile, increasing the concentration of palm stearin in the shortenings was found to decrease TAGs, such as OOO, OOP and OOS but it increased PPO, and vice versa. However, the TAGs containing oleic and palmitic acids, such as OOP and PPO, were observed to be the dominant TAGs present.

Viscosity and viscoelastic measurements

Viscosity can be defined as the resistance to flow liquid. A high viscosity means that the liquid will not flow easily and vice versa. Meanwhile, the viscosity values (Pa.s) versus shear rate (1/s) for the RBD palm oil/palm stearin-based shortenings and commercial shortening are shown in Figure 1. The results revealed that as the shear rate increases, the viscosity decreases, indicating that the flow of shortenings is pseudoplastic.

As expected, the increase in palm stearin in the formulations was found to produce a progressive increase in the viscosity values. This indicates that a high melting temperature TAG present in palm stearin has a significant effect on crystallization and rheological profile of shortenings. A sample containing 100% palm stearin had the highest viscosity among all other samples, whereas the lowest viscosity was shown by the sample containing 100% palm oil.

Fat products, such as shortening, comprise of a fat crystal network that is formed by clusters of solid fat crystal and liquid oil entrapped within. These materials are viscoelastic, and thus they possess both viscous and elastic characteristics (Tang and Marangoni, 2006). Small deformation measurements are usually carried out using dynamic oscillatory controlled stress or controlled strain rheometry. The viscoelastic property of shortenings can be adequately determined using two parameters, namely the storage modulus (G′) which is a measure of its elastic quantity or solid behaviour, and the loss modulus (G″) which is a measure of its viscous quantity or liquid behaviour (Lee et al., 2008a ; Aguirre-Mandujano et al., 2009).

In the present study, the storage modulus (G′) and the loss modulus (G″) were determined as the function of shear stress (Pa) in the RBD palm oil/palm stearin-based shortenings and in the commercial shortening. Among the investigated samples, G′ and G″ parameters were found to increase as the concentration of palm stearin was increased. In this work, the G′ values were always higher than those of G″, indicating that the shortenings were more elastic rather than viscous in character, and were highly structured. The sample containing 80% palm oil and 20% palm stearin (80:20) has the lowest values of G′ and G″, while the highest values were found in the sample containing 100% palm stearin (Figure 2). However, the sample which contained 100% of palm oil (control) did not have storage modulus (G′). This could probably be due to the lower viscosity for this particular sample. These values indicate that the presence of palm stearin has a direct influence on both the elastic and viscous behaviours of the shortenings produced.

Analysis of the cooling and heating thermograms by DSC

Differential scanning calorimetry (DSC) is a technique that is used to measure the function of the differential heat flow with temperature for the compounds showing thermal transitions such as melting and crystallization (Foon et al., 2006). DSC was used in the studies conducted on pure triacyglycerols and triacyglycerol mixtures. The result showed that these components did not crystallize completely in the temperature range of practical interest. Meanwhile, the crystallization of shortenings depicted a positive (exothermic) heat effect, while melting of shortenings had been shown to have caused a negative (endothermic) heat effect.

Peak is an interpretation of change in the differential heat flow, which is caused by the changes in the samples associated with absorption or evolution of heat. There is a direct correlation between the area under the peak and enthalpic change, and this direction will indicate whether the thermal event is endothermic or exothermic (Abdulkarim and Ghazali 2007).

The DSC cooling thermograms of the shortenings are shown in Figure 3. Two distinct peaks, namely major and minor, were observed in the crystallization behaviour. The major peak was found in the high temperature region, i.e. 21-31°C, while the minor peak was observed in the low temperature region of 5-7°C. As previously reported by Chiavaro et al. (2008), as well as Tan and Che Man (2002), the higher temperature exotherm corresponded to the crystallization of the stearin fraction, while the lower temperature exotherm was attributed to the crystallization of the olein fraction. The temperature values of the first major peak of the shortenings showed increment as the concentration of palm stearin was increased.

Meanwhile, the DSC heating thermograms obtained for the shortenings are shown in Figure 4. The samples of shortenings were found to have

completely melted at 43.89-46.53°C, when heated at 5°C/min. Moreover, the two endothermic events could be well distinguishable; these are the low temperature endotherm (olein fraction) and the high temperature endotherm (stearin fraction).

Different formulations of the palm stearin/palm oil-based shortenings showed different melting and crystallization profiles. Based on the figures, it is obvious that the melting and crystallization behaviours of shortenings vary greatly with the increasing amount of palm stearin in the formulations. This finding indicates the importance of palm stearin and how it can be fully exploited for application in food products.

The peak heights of the major crystallization and melting events of shortenings were measured and their values are shown in Figures 5 and 6, respectively. As observed in the findings, the peak heights of the major crystallization increased significantly ($p<0.05$) with the increase in the palm stearin content. Likewise, the peak heights of the major melting event also revealed an increase. However, no significant differences ($p<0.05$) were observed among the samples containing 50:50 and 40:60, 20:80 and 0:100 of the PO:PS ratios.

Conclusions

This study has shown that blending is an effective way to modify the physical and chemical characteristics of the RBD palm oil/palm stearin and their blends. Thus, the palmitic acid (C16:0) and oleic acid (C18:1) were the major fatty acids in these blends. As the concentration of palm stearin was increased, the OOO, OOP and OOS exhibited a decrease, while PPO showed an increase in the content. The highest values of viscosity (Pa), storage modulus (G′) and loss modulus (G″) were shown in those samples containing 100% of palm stearin. The thermal properties such as Onset, Peak, and Endset temperatures (ºC) underwent significant changes due to the increase or decrease of palm stearin content. These findings reveal that crystallization and rheolgical profiles of the shortenings have been significantly affected by the high melting temperature of the TAGs present in palm stearin.

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