

Effect of Chemical Interesterification on the Dropping Point of Palm Oil, Sunflower Oil and Palm Kernel Olein Blends

Noor Lida Habi Mat Dian* and Nor Aini Idris*

ABSTRACT

Changes in the melting points of palm oil (PO), sunflower oil (SFO), palm kernel olein (PKOo) and their blends thereof before and after chemical interesterification (CIE) were studied by the dropping point (DP) method. PO and binary blends of PO/SFO at 3:1 ratio, PO/PKOo at all ratios and ternary blends of PO/SFO/PKOo at 4:1:1, 1:1:4 and 1:1:1 ratios increased in their DP following CIE, indicating that they became harder than their respective non-interesterified (non-IEed) blends. The DPs of PKOo, binary blends of PO/SFO at 1:1 and 1:3 ratios and all binary blends of PKOo/SFO decreased following CIE. The DP of SFO before and after CIE could not be measured as it was very soft even at low temperature. DP data also indicated a eutectic interaction between PO and PKOo in the non-IEed blends of PO/PKOo but that the interaction diminished after CIE.

ABSTRAK

Perubahan dalam takat lebur minyak sawit (PO), olein isirung sawit (PKOo) dan minyak biji bunga matahari (SFO) serta adunan ketiga-tiganya dalam pelbagai nisbah sebelum dan selepas interesterifikasi kimia (CIE) dikaji melalui kaedah takat titisan (DP). PO dan adunan perduaan PO/SFO pada nisbah 3:1, kesemua adunan perduaan PO/PKOo dan adunan pertigaan PO/SFO/PKOo pada nisbah 4:1:1, 1:1:4 dan 1:1:1 mengalami peningkatan dalam DP selepas CIE, menunjukkan bahawa ia adalah lebih keras berbanding dengan adunan langsung (bukan-IEed) masing-masing. DP bagi PKOo, adunan perduaan PO/SFO pada nisbah 1:1 dan 1:3 serta kesemua adunan perduaan PKOo/SFO berkurangan selepas CIE. DP bagi SFO

sebelum dan selepas CIE tidak dapat diukur kerana berbentuk cecair walau pada suhu yang sangat rendah. Data DP juga menunjukkan kehadiran interaksi eutektik di antara PO dan PKOo di dalam adunan langsung PO/PKOo dan interaksi ini berkurangan selepas CIE.

Keywords: blending, chemical interesterification, dropping point, palm kernel olein, palm oil.

INTRODUCTION

Most oils and fats have some limitations in their intended applications because of their triacylglycerol (TAG) and fatty acid (FA) compositions. To widen their use, they can be modified by blending and/or fractionation (physical modifications) or interesterification (IE) and/or hydrogenation (chemical modification) (Petrauskaite *et al.*, 1998). Changing the natural physical characteristics produces an oil and fat with greater functionality for a large number of food product formulations (Rozendaal, 1989; Allen, 1998).

Blending is the simplest modification. However, as the requirements for fat products become more sophisticated, there is correspondingly less likelihood that the specifications can be met simply by blending natural oils and fats. It is very likely that other modification techniques will also be needed for one or more of the blend components in order to meet the product specifications at the lowest practical cost (Allen, 1998). Hydrogenation of polyunsaturated vegetable oils has become less appealing because of the evidence that *trans* FAs have adverse nutritional effects (Mensink and Katan, 1990; Judd *et al.*, 1994). *Trans* FAs raise

* Malaysian Palm Oil Board, P. O. Box 10620, 50720 Kuala Lumpur, Malaysia.

low-density lipoprotein cholesterol and cause arteries to become more rigid and clogged, leading to heart disease. In 2003, Denmark became the first country to introduce restrictions on the use of industrially produced *trans* FAs. The rest of Europe has yet to impose such rules, but pressure is mounting for them to do so from consumer-led organizations (Food Quality news.com, 2004a,b). In the USA, food manufacturers have to include the *trans* FAs content on food labels from 1 January 2006 as required by the new USA Food and Drug Administration rules. Concern over the adverse health implications of *trans* FAs formed during partial hydrogenation has raised interest in alternative ways of producing fats with the required functional properties, yet free from *trans* FAs. The food industry is now relying more on natural solid/semi-solid fats *e.g.*, palm oil (PO) and/or interesterified (IEed) fats to replace partially hydrogenated (*trans*) fats in solid fat food formulations.

PO tends to crystallize in beta prime (β') crystals and is an attractive option for solid fat food formulations. However, PO alone, in its original form, does not result in products that melt quickly in the palate (Moran, 1993). PO also crystallizes more slowly than other fats and oils, leading to the phenomenon of post hardening in which the solid fat products become harder during storage (List, 2004). Hence, PO needs to be blended and/or IEed with other oils and fats to give blends with improved melting and crystallization properties. In solid fat formulations such as table margarine, lauric fats with their broad spectrum of medium chain length FAs are often used as one of the fat blend components to improve the crystallization and melting properties of the end products. In addition, liquid vegetable oils are used as a source of unsaturated FAs and provide lubrication or spreadability to table margarines (Allen, 1998).

IE involves an interchange of FAs within and between the TAG molecules which constitute the fats and oils, and leads to an increased number of TAG types. Thus, the important physico-chemical properties of the fats and oils, such as their melting and crystallization behaviour, are modified. The precise effects of IE on the properties of a fat and oil depend very much on the starting material, or mixture of starting materials that is being IEed. For instance, the melting point and solid fat content (at various temperatures) of a

high melting fat is reduced when it is IEed with a liquid oil (Rozendaal, 1989).

Several IE processes are available with random IE the most commonly used. Random IE is typically carried out at 80°C to 120°C. The reaction is quick with complete randomization usually achieved in only 30-60 min. Directed and 1,3-specific IE are the alternatives to random IE. Directed IE is initiated in exactly the same way as random IE. It differs only in that once the reaction has started, the temperature is reduced to such level that the more saturated TAGs crystallize as they are formed. Once in the solid phase, they take no further part in the reaction. At the end of the reaction, the entire mass is heated to melt the solid portion to yield a homogeneous mixture. As the temperatures used are lower in most directed IE, the reaction is slower and the time taken consequently longer. Careful control of the cooling rates and holding times are essential in ensuring uniform batch production. The 1,3-specific IE is relatively recent. It relies on a 1,3-position specific lipase to catalyze the IE reaction which restricts the FA random interchange only to the outer 1- and 3-positions. The FA in the 2-position is unaffected. Since enzymes are used to catalyze these reactions, the temperatures used are low in comparison to chemically-catalyzed reactions, *i.e.*, approximately 60°C. The reaction is therefore slow and reaction times of 18-24 hr or even longer are typical (Allen, 1998; Husum *et al.*, 2004).

In this study, the effects of blending and random chemical IE (CIE) on the melting behaviour of PO, sunflower oil (SFO), palm kernel olein (PKOo) and blends thereof in various ratios were studied. The melting point is an important physical property of a fat or oil as it determines their suitability in food applications (Timms, 1985). The melting point can be determined by several methods and, in this study, the dropping point (DP) method was adopted. The interaction between PO, SFO and PKOo and their compatibility due to blending and CIE, as shown by their DP properties, are also discussed.

MATERIALS AND METHODS

Materials

PO and SFO were obtained from PGEO Edible Oils Sdn Bhd, Malaysia and PKOo from Cargill Palm Products Sdn Bhd, Malaysia.

Blend Preparation and Chemical Interesterification

The blends preparation and CIE method are as described previously by Noor Lida *et al.* (2002).

Dropping Point

DP was measured according to AOCS Official Method Cc 18-80 (1981) using the Mettler Toledo DP apparatus. The instrument contains a sample cup with a 2.8 mm diameter opening at the bottom. A chilled sample cup was placed on a smooth surface (*e.g.*, glass plate) and molten fat sample (heated at 80°C for 15 min) was poured into it. The sample was allowed to solidify in a -20°C freezer for 15 min, then transferred into the Mettler Toledo DP apparatus which initial temperature was set at least 5°C below the expected DP of the sample. Subsequently, the sample heated at a constant rate of 1°C/min, until it began to flow under its own weight. The temperature at which the sample began to flow through the orifice at the bottom of the cup was its DP.

Statistical Analysis

General linear models and response surfaces analysis of the DP data was performed using Design-Expert Version 6.0.4 (Stat-Ease Inc., Minneapolis, MN). The R^2 values, which indicate the model fits in each of the constructed

ternary diagrams, were determined and found to be greater than 0.97.

RESULTS AND DISCUSSION

Dropping Points of Initial PO, SFO, PKOo and Blends Thereof

The DPs of the initial (non-IEed) and chemically IEed (CIEed) PO (coded A), SFO (coded E), PKOo (coded I) and their blends in various ratios (coded C-D, F-H and J-Q) are presented in *Table 1*. The percentage of reduction or increment in the DP are also presented. Of the three oils, PO had the highest DP due to its high content of long chain saturated FAs, *i.e.*, palmitic and stearic acids, followed by PKOo and SFO. Since SFO is a liquid oil containing high amounts of monounsaturated and polyunsaturated FAs, its DP could not be measured using this method. Bernardini (1983) stated that the higher the degree of unsaturation of a FA, the lower the melting point (in this case, the DP) of the TAG it constitutes as the melting point of a TAG is related to those of its constituent FAs. Though PKOo, a lauric-rich fat, can be considered a saturated fat (78% saturated FAs), it has a lower DP than PO because of the types of its FAs. The majority of FAs in PKOo are medium and short chain saturates, *i.e.*, lauric, myristic, caprylic and capric, which have lower melting

TABLE 1. DROPPING POINTS OF PALM OIL (PO), SUNFLOWER OIL (SFO), PALM KERNEL OLEIN (PKOo) AND THEIR BLENDS IN VARIOUS RATIOS BEFORE (DB) AND AFTER (CIE) CHEMICAL INTERESTERIFICATION^a

Code	PO/SFO/PKOo ratio	Dropping point (°C)		% Increase or reduction after CIE
		DB	CIE	
A	1:0:0	37.6	42.5	13.0 ⁺
B	3:1:0	35.5	35.8	0.8 ⁺
C	1:1:0	30.2	24.9	17.5 ⁻
D	1:3:0	13.7	8.5	38.0 ⁻
E	0:1:0	Liquid	Liquid	Unchanged
F	0:3:1	11.5	6.5	43.5 ⁻
G	0:1:1	17.4	13.5	22.4 ⁻
H	0:1:3	24.3	19.4	20.2 ⁻
I	0:0:1	26.2	25.5	2.7 ⁻
J	1:0:3	24.2	26.7	10.3 ⁺
K	1:0:1	23.6	28.5	20.8 ⁺
L	3:0:1	32.0	35.3	10.3 ⁺
M	4:1:1	32.4	33.8	4.3 ⁺
N	1:4:1	11.5	10.3	1.7 ⁻
P	1:1:4	23.8	24.5	2.9 ⁺
Q	1:1:1	18.6	22.5	21.0 ⁺

Note: ^aSuperscript (+) or (-) after each figure indicates an increase or decrease, respectively.

points than the longer chain saturated FAs of PO. The FA compositions of PO, SFO, PKOo and their blends in various ratios are shown in Table 2.

The DPs of PO/SFO and PKOo/SFO blends gradually decreased with an increase in the proportion of SFO. The softening effect was due to the liquid triunsaturated (U_3) TAGs in SFO diluting the solid disaturated-monounsaturated (S_2U) and trisaturated (S_3) TAGs in PO and PKOo. The blends of PO/SFO and PKOo/SFO had DPs representing linear combinations of the two components. On the other hand, the DP of the PO/PKOo blends did not represent linear combinations of PO and PKOo. The DPs of the blends were lower than both those of PO and PKOo. According to Dieffenbacher (1988), if the physical characteristics of components in a fat blend do not represent linear combinations of their components, it is indicative of some interaction between the components. In this case, the interaction between PO and PKOo was eutectic. An eutectic interaction is often observed in fat mixtures. This interaction tends to occur when the fats differ in molecular volume, shape or polymorph. A mixture with eutectic effect will have a lower melting point than either of the two pure fats, showing the incompatibility between the two fats (Bigalli, 1988; Timms, 1984). Eutectic effects are usually undesirable, but in margarines and shortenings, the effect can be beneficial (deMan and deMan, 1994). The PO/PKOo blend reached its minimum DP, or maximum eutectic effect, at about 1:1 blending ratio (Figure 1). The eutectic interaction in binary systems of PO and PKOo had been previously reported by Timms (1985).

The DPs of ternary blends of PO/SFO/PKOo varied, depending on the amounts of PO, SFO and PKOo present. Thus, the blend with the highest proportion of PO (coded M) had the highest DP, followed by the blend containing a high proportion of PKOo (coded P) and the blend containing equal proportions of each lipid (coded Q) (Table 1). As expected, the ternary blend with the highest proportion of SFO (coded N) had the lowest DP. The DP of blend M was about 8.6 (26.5%), 13.8 (42.6%) and 20.9°C (64.5%) higher than those of blends P, Q and N, respectively.

Dropping Point of Chemically Interesterified PO, SFO, PKOo and their Blends Thereof

PO increased in DP upon CIE due to a rise in its S_3 TAGs, *i.e.*, PPP and PPSt, where P = palmitic, and St = stearic acids, and a significant decrease in some S_2U (PLP, POP and POST) and diunsaturated-monosaturated (U_2S) (POO and StOO) TAGs, where L = linoleic, and O = oleic acids. The PPP and PPSt of PO increased from 6.7% and 1.1% before CIE to 10.0% and 2.5%, respectively, after CIE. On the other hand, PLP, POP, POST, POO and StOO were reduced from 9.2%, 30.2%, 6.7%, 23.3% and 2.9% before CIE to 6.6%, 26.9%, 5.4%, 22.5% and 2.1%, respectively, after CIE (Noor Lida *et al.*, 2002). These results are consistent with Allen (1998) who stated that most IEed oils and fats have higher melting points, almost certainly due to the formation of S_3 TAGs. There was no change in the DP of SFO after CIE. This was presumably because SFO is very rich in unsaturated long chain FAs, and therefore, randomization did not result in any great change in its TAG composition. As a result, its

TABLE 2. FATTY ACID COMPOSITION OF PALM OIL (PO), SUNFLOWER OIL (SFO) AND PALM KERNEL OLEIN (PKOo)^a

Oil	FAC (wt %)								
	C _{8:0}	C _{10:0}	C _{12:0}	C _{14:0}	C _{16:0}	C _{18:10}	C _{18:1}	C _{18:2}	Others
PO	–	–	0.2	1.0	42.9	4.4	40.8	10.2	0.5
SFO	–	–	–	0.1	6.3	3.7	24.3	65.1	0.5
PKOo	47	3.8	44.5	13.7	8.4	2.8	18.1	2.9	1.1

Note: ^aFAC, fatty acid composition; C_{8:0}, caprylic acid; C_{10:0}, capric acid; C_{12:0}, lauric acid; C_{14:0}, myristic acid; C_{16:0}, palmitic acid; C_{18:0}, stearic acid; C_{18:1}, oleic acid; C_{18:2}, linoleic acid. Others include caproic (C_{6:0}) and/or linolenic (C_{18:3}) and/or arachidic (C_{20:0}) acids.

Source: Adapted from Noor Lida *et al.* (2002).

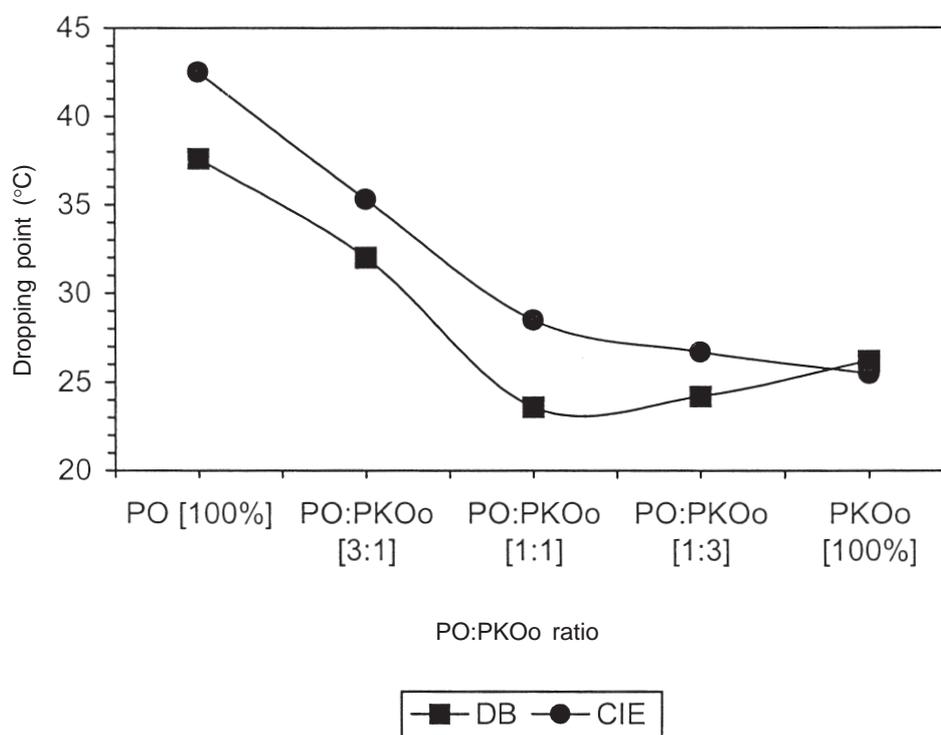


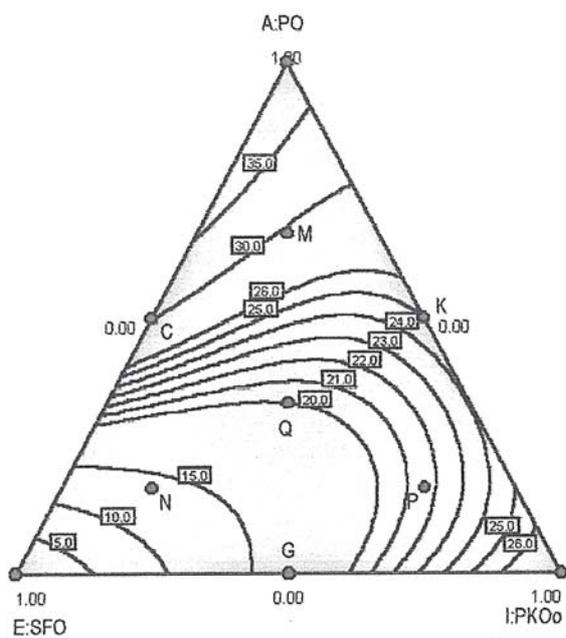
Figure 1. Dropping points of palm oil (PO) and palm kernel olein (PKOo) blends in various ratios before (DB) and after (CIE) chemical interesterification.

DP was hardly altered. CIE slightly reduced the DP of PKOo. This could have been due to decreases in some of the S_3 TAGs, e.g., CLaLa, CaLaLa/CLaM and LaLaLa, where C = capric, La = lauric, Ca = caprylic, and M = myristic acids, and increases in the TAGs species with intermediate degrees of unsaturation, e.g., LaLaO and LaOM. CLaLa, CaLaLa/CLaM and LaLaLa were reduced from 8.4%, 11.8% and 22.7% before CIE to 5.5%, 6.6% and 18.6%, respectively, after CIE. On the other hand, the LaLaO and LaOM were increased from 6.4% and 5.0% before CIE to 13.6% and 7.6% after CIE (Noor Lida *et al.*, 2002).

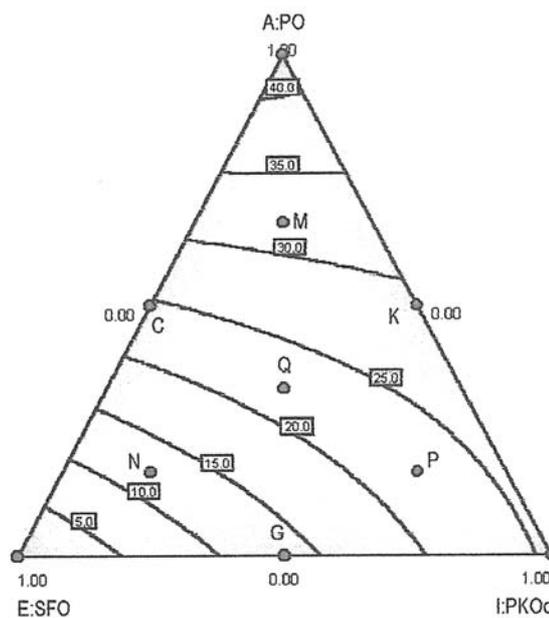
All the binary blends of PO/SFO, PKOo/SFO and PO/PKOo had DPs representing linear combinations of their two components following CIE. This indicated that the eutectic interaction between PO and PKOo had been eliminated (Figure 1) by CIE. Elimination of the eutectic interaction demonstrates a better miscibility between PO and PKOo after CIE, is shown by the isosolid diagram in Figure 2. Generally, the DP reductions by the CIEed binary blends due to the increased proportion of SFO (for the binary blends of PO/SFO and PKOo/SFO) and PKOo (for the binary blends of PO/PKOo) were larger than the reductions observed in their respective non-IEed blends (Figure 3). For

instance, the DP for the CIEed blend coded D (75% SFO) was 12.1°C (43.7%) lower than that for the blend coded C (50% SFO). For the non-IEed blend, only a 7.1°C (23.5%) reduction was observed.

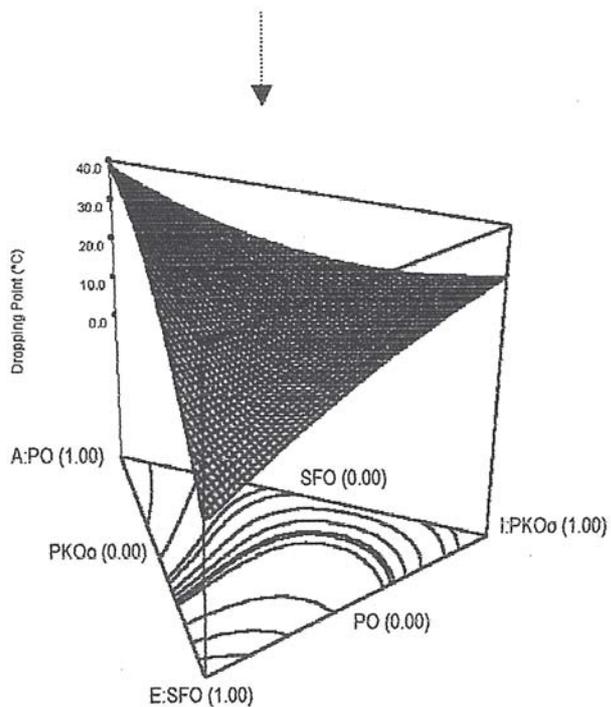
The binary blends of PO/SFO, PKOo/SFO and PO/PKOo also underwent remarkable changes in their DPs following CIE. The changes in the DPs of PO/SFO and PKOo/SFO blends were dependent on the proportion of SFO incorporated. The higher the proportion of SFO incorporated, the greater was the difference in DPs. This is in agreement with Petrauskaitė *et al.* (1998), Ghosh and Bhattacharyya (1997) and Forsell *et al.* (1992). The PO/SFO blend containing 25% SFO (coded B) hardly changed following CIE with only a 1.4% increase in its DP compared to 100% PO which showed a 13.0% increase. The PKOo/SFO blend containing the same amount of SFO (coded H) had a 6.6% reduction in its DP compared to only a 1.1% reduction in 100% PKOo. The blends of PO/SFO and PKOo/SFO containing 50% SFO (coded C and G, respectively) showed 8.2% and 16.6% reductions in their DPs, respectively. As expected, the greatest reduction in DP was observed in the blends of PO/SFO and PKOo/SFO containing 75% SFO (coded D and F, respectively). They had 32.5% and 30.9%



Contour

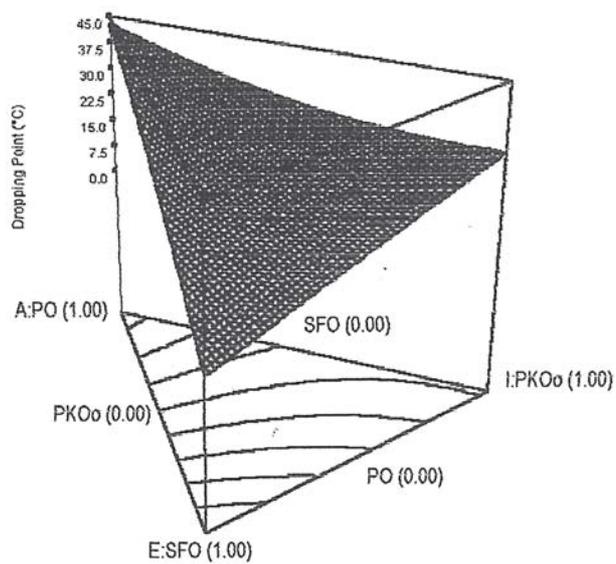


Contour



3D Surface

(2a)



3D Surface

(2b)

Figure 2. Contour and 3D surface diagrams of dropping points of palm oil (PO), sunflower oil (SFO) and palm kernel olein (PKOo) ternary blends before (a) and after (b) chemical interesterification.

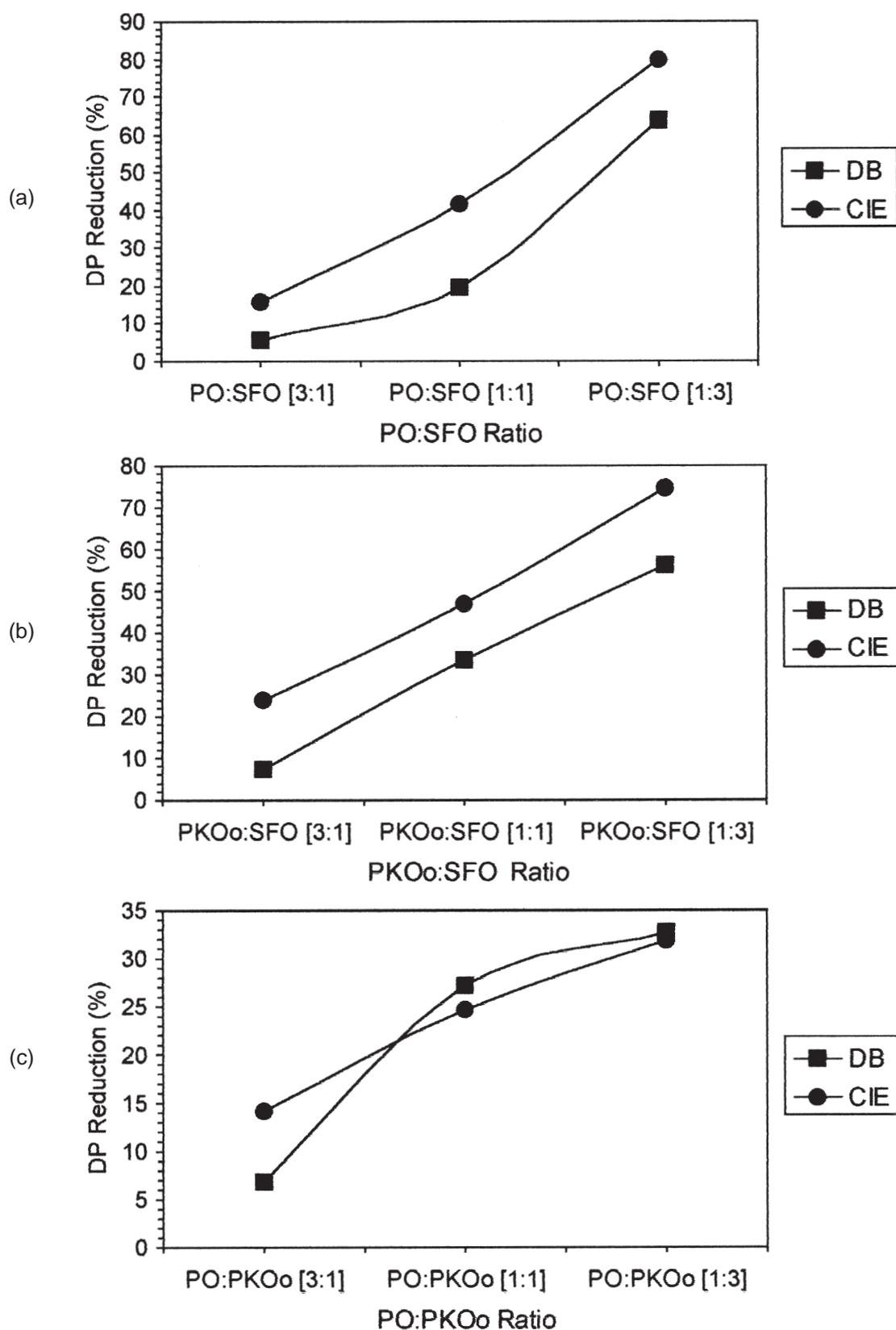


Figure 3. Dropping point (DP) reduction of the binary blends of (a) palm oil (PO) and sunflower oil (SFO), (b) palm kernel olein (PKOo) and SFO and (c) PO and PKOo from addition of SFO and PKOo before (DB) and after (CIE) chemical interesterification.

reductions in their DPs, respectively. The slight increase in DP experienced by blend B was presumably due to the slight increase in the very high melting S_3 TAG, *i.e.*, PPSt. PPSt increased from 0.8% before CIE to 1.3% after CIE. The melting point of PPSt is 68°C (Timms, 1984). The decreases in the DP of the CIEed PO/SFO blends coded C and D were mainly attributed to decreases in their proportions of high melting S_3 and S_2U TAGs, mainly PPP (S_3) and POP (S_2U), concomitant with the formation of more U_2S TAGs such as PLL and PLO. For instance, PPP and POP in blend coded C decreased from 3.5% and 16.0% before CIE to 1.3% and 8.9% after CIE. On the other hand, PLL and PLO increased from 5.5% and 10.1% before CIE to 10.0% and 21.3% after CIE (Noor Lida *et al.*, 2002). For the PKOo/SFO blends, the reductions in their DP were most likely due to decreases in their proportions of S_3 medium chain TAGs, such as LaLaLa, CaLaLa, CLaLa and LaLaM, simultaneous with the formation of several species of low melting TAGs. The formation of low melting TAGs in the CIEed binary blends of PO/SFO and PKOo/SFO was due to replacement of the saturated FAs in the TAGs of PO and PKOo with the unsaturated FAs of SFO TAGs, which were mainly U_2S and U_3 (Noor Lida *et al.*, 2002).

All the binary blends of PO/PKOo underwent noteworthy increases in DP following CIE although their proportions of high melting S_3 and S_2U TAGs, such as LaLaLa, PPP and POP, were hardly altered. The increases were due to elimination of the eutectic interaction between PO and PKOo. According to Timms (1984), IE would eliminate or, at least, reduce the eutectic interaction in a eutectic mixture. Due to their incompatibility, the non-IEed blends of PO/PKOo were much softer than they should be. In the ternary blends of PO/SFO/PKOo, only modest changes in their DP were observed following CIE.

CONCLUSION

CIE altered, and, in most cases, reduced the DPs of PO/SFO/PKOo blends. CIE also improved the miscibility between PO and PKOo. PO, when CIEed with an oil of shorter chain FAs (*e.g.*, PKOo) and/or oil rich in polyunsaturated FAs (*e.g.*, SFO), gave products with satisfactory DPs for various food applications. With CIE, a higher percentage of PO can be incorporated in solid fat food products such as bakery and table margarines and shortenings.

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