

EFFECT OF DIOLS AS CO-SURFACTANTS IN PARTIAL TERNARY PHASE BEHAVIOUR OF PALM OIL-BASED MICROEMULSIONS

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ABSTRACT

Microemulsions (μ E) were prepared with palm fatty acid methyl esters (PFAME) as solvents, and stabilised by ethoxylated non-ionic surfactants, i.e., Dehydol LS 7 and Dehydol LS 2 as S_A and S_B with the hydrophilic-lipophilic balance (HLB) values at ~ 12.8 and ~ 7.3 , respectively. The co-surfactants chosen were 1,2-hexanediol, 1,2-butanediol, 1,2-ethanediol and glycerol. The effects of the co-surfactants on the partial ternary phase diagrams were studied at 10%, 15%, 20%, 25%, 30% and 40% (w/w) concentrations. The ratio of PFAME to water was either 25:75 or 50:50. The mixtures were agitated vigorously, and then kept at ambient temperature (25°C) for one to two days. The emulsions (two-phase), the μ E and liquid crystalline phases were observed using polarised light. In addition, the physical stability of the μ E solutions was determined at 45°C over a period of one month. The effects of the co-surfactants on the ternary phase systems for PFAME/non-ionic surfactants/water were studied and mapped out at 25°C and 45°C, respectively.

Keywords: household and industrial applications, non-toxic co-surfactants, microemulsions (μ E), palm fatty acid methyl esters.

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INTRODUCTION

A microemulsion is a monodisperse solution, which is isotropic (or clear) and thermodynamically stable, in which substantial amounts of two immiscible liquids (i.e., water and oil) are brought into a monodisperse solution by means of an appropriate surfactant or surfactant mixture, with or without a co-surfactant (Hoar and Schulman, 1943; Prince, 1977a). Microemulsions formed with low molecular weight aliphatic or aromatic hydrocarbons have been extensively studied, and data on their compositions, properties and structure have been reported (Alander and Warenheim, 1989). However, there is growing interest in replacing the aromatic hydrocarbon oils with oils derived from natural resources (Alander and Warenheim, 1989; Joubran *et al.*, 1993; Hamdan *et al.*, 1995; Monig *et al.*, 1996;

von Corswant and Soderman, 1998; Alany *et al.*, 2000; Warisnoicharoen *et al.*, 2000; Raman *et al.*, 2003; 2005; 2008). Natural oils and their derivatives, such as long- and medium-chain triglycerides (LCT and MCT) and fatty acid esters (FAE) are renewable, biodegradable, non-flammable, harmless to the environment and less or non-toxic to end-users (Leysen, 1992; Hamilton, 1993; Haigh, 1995; Srivastava and Prasad, 2000).

In this study, palm fatty acid methyl esters (PFAME) were chosen as the oil phases because PFAME have good potential to be utilised as natural-based oil phases or solvents in agrochemicals (Ismail *et al.*, 2000b; 2004), household and industrial cleaners (Aishah, 2004; Choo, 2004a, b; Yeong, 2006), biodiesels (Choo, 1994; 2002), lubricants (Choo, 1995; Loh, 2006) and for other household and industrial applications.

The formation of a stable microemulsion (μ E) often requires a high concentration of surfactant, but this can sometimes be reduced by using a co-surfactant, e.g. 1-pentanol (Prince, 1977b; Overbeek *et al.*, 1984; Cavalli *et al.*, 1996; Ismail *et al.*, 2000a; Raman *et al.*, 2003). However, 1-pentanol and other short-chain alcohols can cause skin and eye irritation. Investigations carried out by several researchers (e.g., Kahlweit, 1995; Kahlweit *et al.*,

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1996; Comelles, 1999; Alany *et al.*, 2000) have found that 1,2-alkanediols have properties similar to aliphatic alcohols, but cause less medical problems to end-users.

This study aims to investigate the effects of 1,2-alkanediols (1,2-hexanediol, 1,2-butanediol, 1,2-ethanediol) and glycerol as non-toxic co-surfactants when used to prepare palm-based μ E. Also, the study focuses on the concentrations of surfactants, in the range of 10% to 40% (w/w), that can be applied in preparing microemulsions for practical applications. The μ E can then be used as μ E-spray insecticides, μ E all-purpose liquid cleaners, μ E body-spray insect repellent, and for other household and industrial applications.

MATERIALS AND METHODS

Materials

PFAME were supplied by Carotech (Malaysia) Sdn Bhd. Ethoxylated non-ionic surfactants, *i.e.* Dehydrol LS 7 as S_A and Dehydrol LS 2 as S_B with hydrophile-lipophile balance (HLB) values of ~ 12.8 and ~ 7.3 , respectively, were provided by Emery Oleochemicals (M) Sdn Bhd [formerly known as Cognis Oleochemicals (M) Sdn Bhd]. The 1,2-hexanediol, 1-pentanol, 1,2-butanediol, 1,2-ethanediol and glycerol were bought from Sigma-Adrich (M) Sdn Bhd.

Methods

The basic mixtures were prepared with 10%, 15%, 20%, 25%, 30% and 40% (w/w) of the ethoxylated non-ionic surfactants and variable concentrations of 1,2-alkanediols and glycerol as the co-surfactants. The ratios of oil to water used were 25:75 and 50:50. Filtered 0.01 M NaCl solution was used as the aqueous phase for preparing the mixtures. The mixtures were agitated vigorously by using a vortex mixer, and incubated in a water-bath for about 2 hr at 50°C. The mixtures were again agitated vigorously and then equilibrated for two days at around 25°C (or ambient temperature), and finally at 45°C for a month.

The formation of microemulsions and other phases (emulsions or liquid crystals) was observed qualitatively using polarised light sheets. The phases observed were mapped out on triangular phase diagrams according to Broze (1997).

RESULTS AND DISCUSSION

The effects of 1,2-alkanediols (1,2-hexanediol, 1,2-butanediol, 1,2-ethanediol) and glycerol as non-toxic co-surfactants on the partial ternary

phase diagrams of palm-based microemulsion (μ E) systems were studied and discussed.

Partial Ternary Phase Diagram of PFAME/Water/Dehydrol LS 7 (S_A) and LS 2 (S_B)/1,2-Hexanediol

The screening process with 20% (w/w) of Dehydrol LS 7 (S_A) showed that PFAME-microemulsions were formed at 15% and 25% (w/w) of 1,2-hexanediol for the 25:75 and 50:50 ratios of oil to water at both 25°C and 45°C. With 20% (w/w) of mixed Dehydrol LS 7 (S_A)/Dehydrol LS 2 (S_B), PFAME-microemulsions formed at 15% (w/w) of 1,2-hexanediol for both the 25:75 and 50:50 ratios of oil to water at 25°C, and at 15% and 10% (w/w) of 1,2-hexanediol for both oil to water ratios at 45°C. These results indicate that the miscibility of PFAME and water (at both ratios) in mixed S_A/S_B solutions was slightly higher than in S_A solution when 1,2-hexanediol was added as the co-surfactant. Furthermore, the formation of microemulsions was better with a 25:75 than with a 50:50 ratio of PFAME to water in S_A , but was about equivalent with both ratios in mixed S_A/S_B solution. This means that the mixed S_A/S_B solution had a slightly higher capacity to solubilise PFAME and water than the S_A solution, thus leading to the formation of microemulsion solutions.

Figures 1 and 2 show the partial ternary phase diagrams of the surfactant S_A and the S_A/S_B surfactant mixture at a 75:25 ratio with 1,2 hexanediol (a conventional co-surfactant), respectively. The ratio of oil to water in both systems was 25:75. The diagrams determined that the minimum concentration to form microemulsions was 10% (w/w) for both systems. However, the concentrations of 1,2-hexanediol required to form microemulsions with 10% (w/w) of both S_A and mixed S_A/S_B solutions were 30% and 20% (w/w), respectively.

The optimum concentrations of S_A and 1,2-hexanediol (Figure 1) to form microemulsions were 20% and 15% (w/w), respectively, at both 25°C and 45°C. However, the optimum concentrations for mixed S_A/S_B and 1,2-hexanediol (Figure 2) were 20% and 10% (w/w), respectively, at both 25°C and 45°C. In addition, there was a three-phase region observed in PFAME/mixed S_A/S_B (75:25 ratio)/1,2 hexanediol/water at 45°C (Figure 2b). This three-phase region was a bicontinuous (or middle-phase) microemulsion containing comparable amounts of water and oil phases coexisting with both excess of oil and water.

The effect of 1,2-hexanediol as a (non-toxic) co-surfactant on the formation of PFAME-microemulsions was also compared to the effect of 1-pentanol, the medium-chain aliphatic alcohol (Figure 3) (Raman *et al.*, 2005; Ismail, 2006). The microemulsion regions formed with the

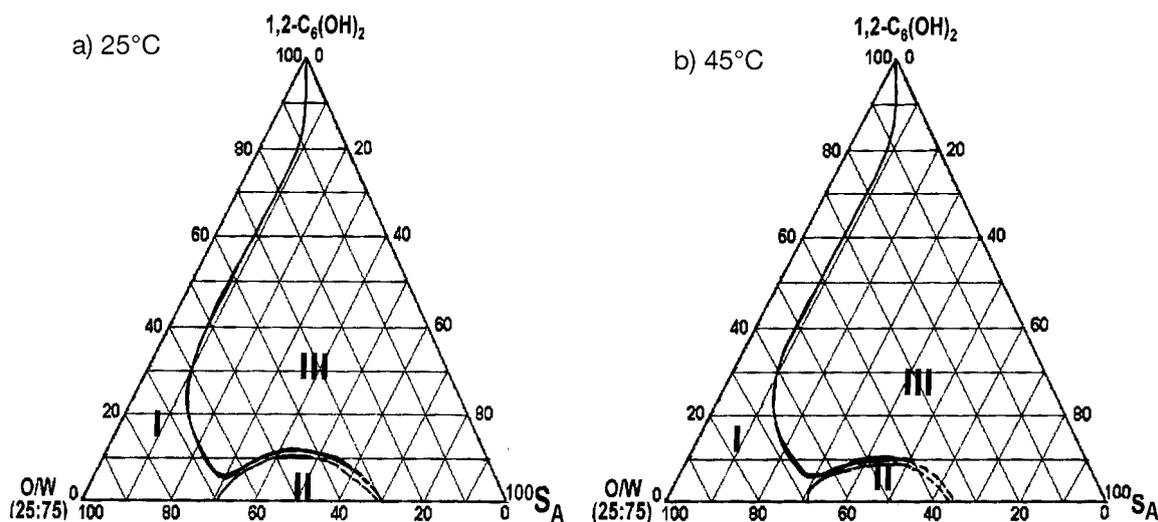


Figure 1. Effects of 1,2-hexanediol [or 1,2- $C_6(OH)_2$] on the partial ternary phase diagrams of S_A solution and 25:75 of PFAME/ H_2O at (a) 25°C and (b) 45°C. I, II, and III are the two/multiple-phase (or emulsion), liquid crystalline and O/W and/or W/O microemulsion regions, respectively. The S_A and O/W are defined as the surfactant and the ratio of oil to water, respectively.

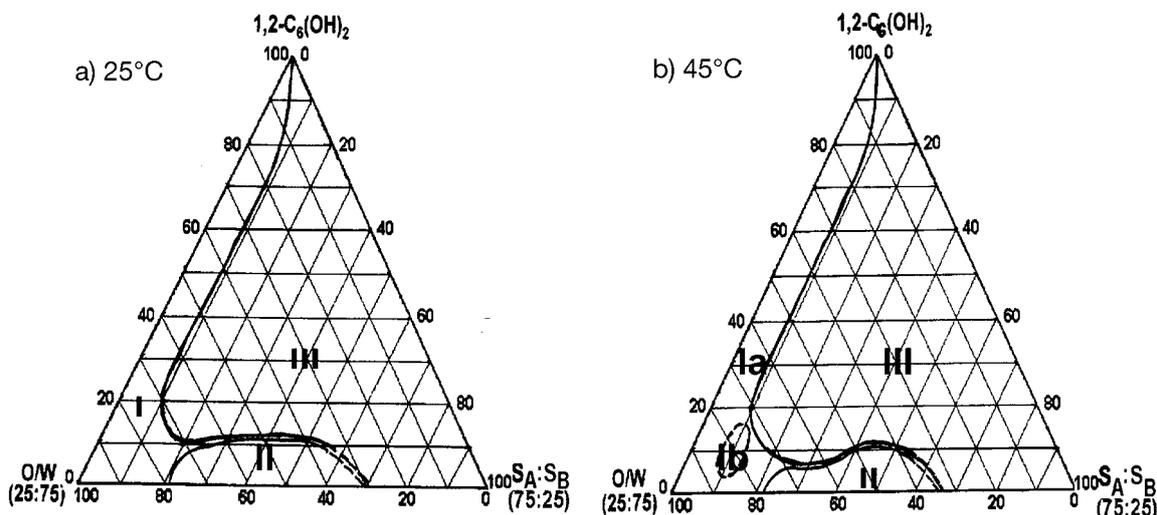


Figure 2. Effects of 1,2-hexanediol [or 1,2- $C_6(OH)_2$] on the partial ternary phase diagrams of 75:25 of S_A/S_B and 25:75 of PFAME/ H_2O at (a) 25°C and (b) 45°C. I, II and III are two/multiple-phase (or emulsion), liquid crystalline, and O/W and/or W/O microemulsion regions, respectively. The S and O/W are defined as the surfactant and the ratio of oil to water, respectively.

PFAME/surfactants (S_A and/or mixed S_A/S_B)/1,2 hexanediol/water) (Figures 1 and 2) were larger than the PFAME/surfactant S_A /1-pentanol/water system (Figure 3). These results show that S_A and/or mixed S_A/S_B (at a 75:25 ratio) with the addition of 1,2-hexanediol had higher solubilisation capacity for both PFAME and water than the S_A /1-pentanol system.

Triangular Phase Diagram of PFAME/Water/Dehydrol LS 7 (S_A) and LS 2 (S_B)/1,2-Butanediol

The screening of four types of combination mixtures showed that the mixture of Dehydrol LS 7 (S_A)/Dehydrol LS 2 (S_B) at 75:25 ratio together with 1,2-butanediol gave rise to many one-phase microemulsion solutions. Figure 4 illustrates the

partial ternary phase diagrams of S_A/S_B surfactant mixture at 75:25 ratio with 1,2 butanediol as another alternative non-toxic co-surfactant in the system. The optimum concentrations of the S_A/S_B surfactant mixture and 1,2 butanediol were 20% and 10% (w/w), respectively, at 25°C (Figure 4). When the temperature was increased to 45°C, a one-phase microemulsion solution was formed at 20% (w/w) of S_A/S_B , but with 10% (w/w) and/or less of 1,2-butanediol.

A small portion of the lamellar liquid crystals (LC) was destabilised or dissociated and formed a micellar solution and/or O/W microemulsion. Thus, the microemulsion region was enlarged a little at 45°C. The three-phase region (Ib) was observed with PFAME/mixed S_A/S_B (75:25 ratio)/1,2-butanediol/water at 45°C (Figure 4b). However,

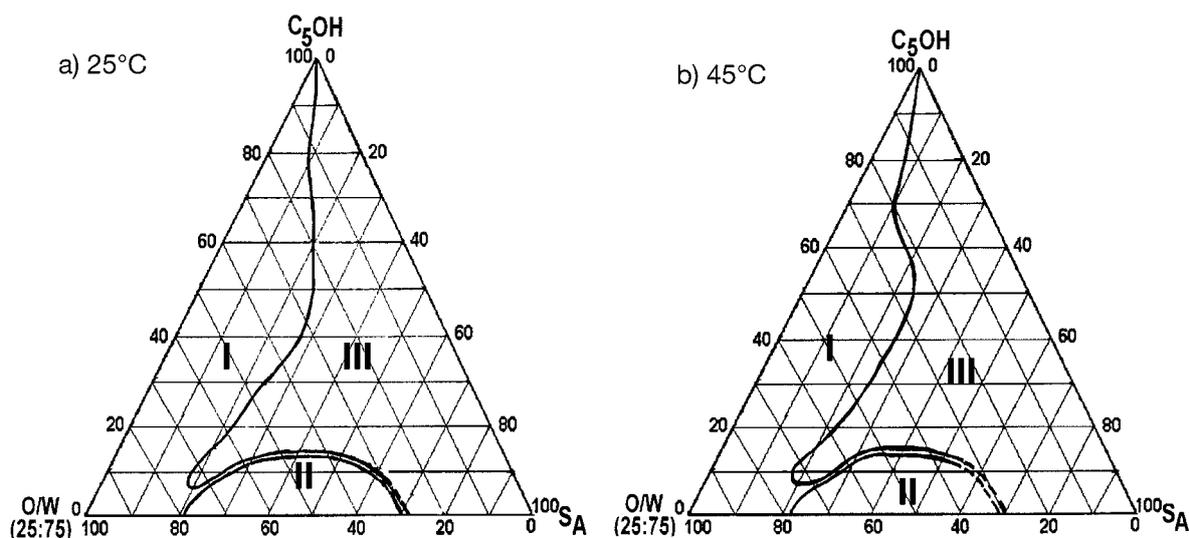


Figure 3. Effects of 1-pentanol (or C_5OH) as co-surfactant on Dehydol LS 7EO/PFAME/water system at (a) 25°C and (b) 45°C. The O/W and S_A refer to oil ratio to water and surfactant, respectively. The O/W ratio is 25:75 and I, II and III represent the emulsion (two/three-phase), liquid crystalline (LC) and microemulsion regions, respectively.

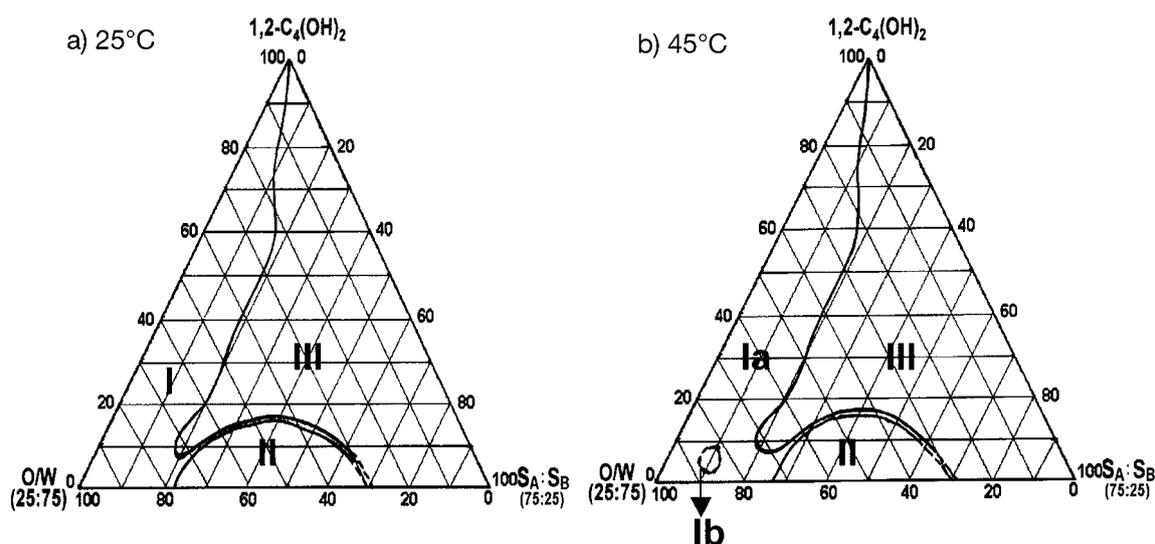


Figure 4. Effects of 1,2-butanediol [or $1,2-C_4(OH)_2$] on the partial ternary phase diagrams of 75:25 of S_A/S_B and 25:75 of POME/ H_2O at (a) 25°C and (b) 45°C. Ia, Ib, II, and III are the two- and three-phase (or emulsion), liquid crystalline and O/W and/or W/O microemulsion regions, respectively. The S and O/W defines the surfactant and the ratio of oil to water, respectively.

this three-phase region was formed only at 10% of mixed S_A/S_B and 5% (w/w) of 1,2-butanediol.

The microemulsion region for PFAME/mixed S_A/S_B /1,2-butanediol/water (Figure 4) was smaller than those formed with PFAME/ S_A /1,2-hexanediol/water (Figure 1) and with PFAME/mixed S_A/S_B /1,2-hexanediol/water (Figure 2). However, the PFAME/mixed S_A/S_B /1,2-butanediol/water system formed a slightly larger microemulsion region than the PFAME/surfactant S_A /1-pentanol/water system (Figure 3) (Raman *et al.*, 2005; Ismail, 2006). These results show that the mixed S_A/S_B (at 75:25 ratio) with addition of 1,2 butanediol had

a lower capacity to solubilise both PFAME and water than 1,2-hexanediol, but had a comparable capacity to solubilise PFAME and water compared to 1-pentanol.

In addition, these results indicate that 1,2-hexanediol had a better synergistic effect as a co-surfactant than 1,2-butanediol to single or mixed non-ionic surfactant systems for lowering the interfacial tension and/or the curvature between the oil and aqueous phases (Alany *et al.*, 2000; Warisnoicharoen *et al.*, 2000). Thus, it had a higher solubilisation capacity to PFAME/water and formed larger microemulsion regions.

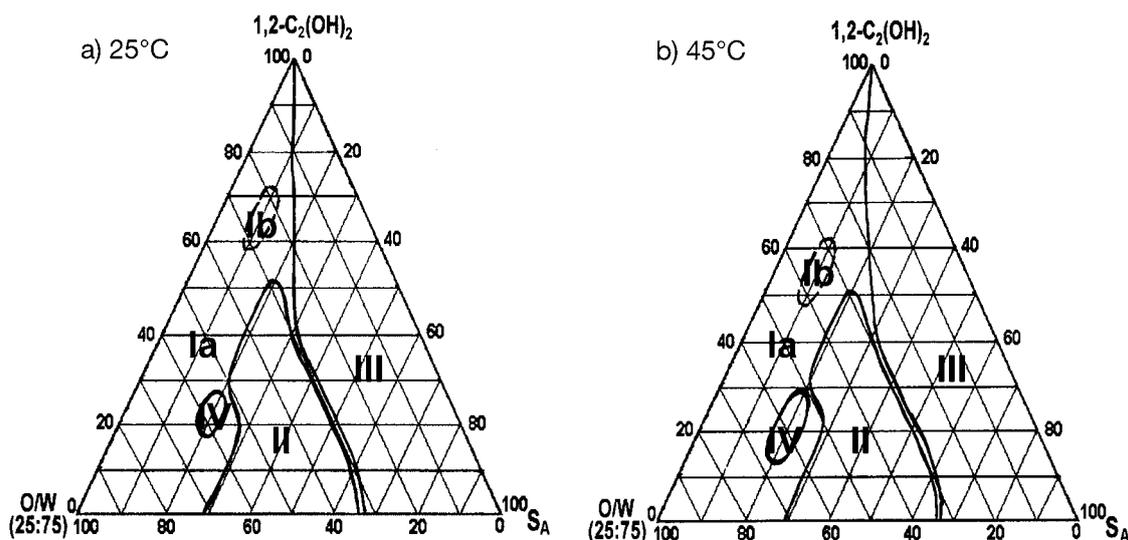


Figure 5. Effects of 1,2-ethanediol [or 1,2-C₂(OH)₂] on the partial ternary phase diagrams of S_A solution and 25:75 of PFAME/water at a) 25°C and b) 45°C. The notations I(a,b), II, III, and IV are emulsion (or two- and three-phase), liquid crystalline, inversed micellar or W/O microemulsion and normal micellar or O/W microemulsion regions, respectively.

Triangular Phase Diagram of PFAME/Water/Dehydol LS 7 (S_A)/1,2-Ethanediol

The screening process also found that one-phase microemulsion solutions were formed at 20% (w/w) of Dehydol LS 7EO (S_A), but only with 15% to 25% (w/w) of 1,2-ethanediol for a 25:75 ratio of PFAME/water. Studies were also carried out with 20% (w/w) S_A solutions at a 50:50 ratio of PFAME to water, and with 75:25 of S_A/S_B for both ratios of oil to water. However, the other mixtures showed two or more layers of heterogeneous solutions.

Figure 5 shows the effects of 1,2-ethanediol on the partial ternary phase behaviour of S_A solutions containing a 25:75 ratio of PFAME to water. The system had larger regions for the two-phase [or emulsion] (Ia), liquid crystalline (II) and the inversed micellar and/or W/O microemulsion (III) regions. However, the system had only a small region for the normal micellar and/or O/W microemulsion (IV), in which the optimum concentrations for surfactant and co-surfactant were 20% and 15% (w/w), respectively, at both 25°C and 45°C. In addition, a three-phase region (Ib) was also observed at 10% (w/w) of S_A solution, but with rather high concentrations of 1,2 ethanediol at both temperatures as shown in Figure 5.

The results above indicate that 1,2-ethanediol required higher HLB values of surfactant or mixed surfactants in the system than 1,2-butanediol and 1,2-hexanediol for the formation of PFAME-microemulsions. Furthermore, they show that S_A and/or mixed S_A/S_B (at 75:25 ratio) with the addition of 1,2-ethanediol had a lower capacity for solubilisation for both PFAME and water than 1,2-butanediol and 1,2-hexanediol. Thus, the system

formed narrow regions of normal micellar solution and/or O/W microemulsion (IV) as illustrated in Figure 5.

Triangular Phase Diagram of PFAME/Water/Dehydol LS 7 (S_A)/Glycerol

The screening process found that a one-phase microemulsion was only formed at 20% (w/w) of Dehydol LS 7EO (S_A) and at 15% of glycerol for the 25:75 ratio of PFAME to water. Studies were also carried out with 20% (w/w) S_A solution at the 50:50 ratio of PFAME to water, and with 75:25 of S_A/S_B for both ratios of oil to water. The mixtures gave two- or three-phase systems (or emulsions), liquid crystals, or microemulsions. However, the diagrams for these regions were not constructed and discussed because the systems did not produce an optimum triangular phase diagram of the microemulsion region.

The partial ternary phase diagram for PFAME/S_A/glycerol/water as shown in Figure 6 was about equivalent to the partial ternary diagram for PFAME/S_A/1,2-ethanediol/water (Figure 5). However, this system had slightly larger emulsion (Ia) and liquid crystalline (II) regions, but a slightly smaller W/O microemulsion (III) region than the PFAME/S_A/1,2-ethanediol/water system. The system also had a small region for the normal micellar and/or O/W microemulsion (IV), in which the optimum concentrations for surfactant and co-surfactant were 20% and 15% (w/w), respectively, at both 25°C and 45°C (Figure 6). In addition, a three-phase region (Ib) was observed at 10% to 20% (w/w) of S_A solution, and ~30%-40% (w/w) of glycerol as shown in Figure 6.

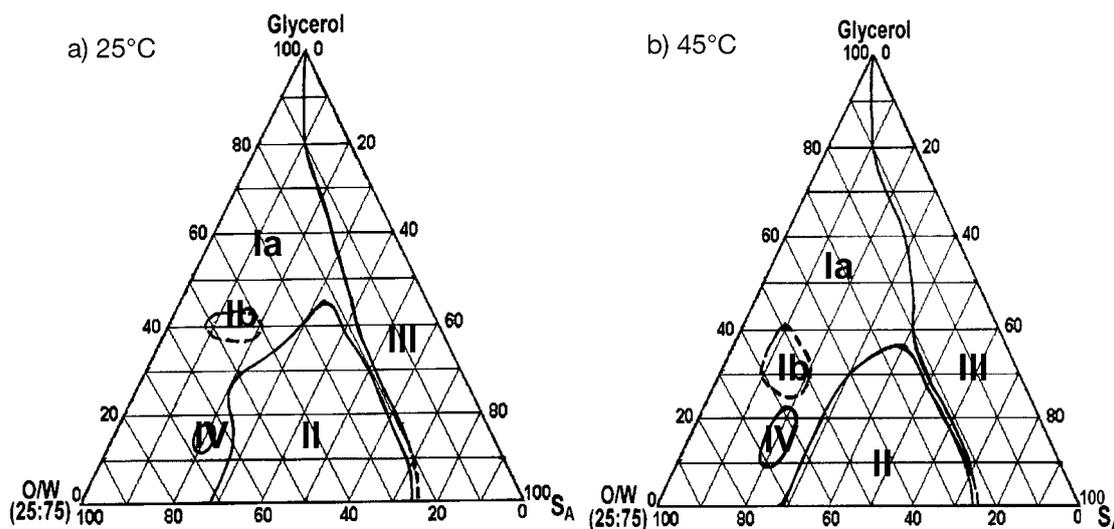


Figure 6. Effects of glycerol on the partial ternary phase diagrams of S_A solution and 25:75 of PFAME/water at a) 25°C and b) 45°C. The Ia,b, II, III, and IV represent the two- and three-phase (or emulsion), liquid crystalline, and W/O and O/W microemulsion regions, respectively. The S and O/W define the surfactant and the ratio of oil to water, respectively.

CONCLUSION

The 1,2-alkanediols (*i.e.*, 1,2-hexanediol, 1,2-butanediol and 1,2-ethanediol) and glycerol show good potential as non-toxic co-surfactants to replace medium-chain aliphatic alcohols such as 1-pentanol in palm-based microemulsions because they cause very minimal irritation to the skin and eyes of end-users, and are less toxic to aquatic organisms. The 1,2-hexanediol has shown better synergistic effects as a co-surfactant than 1-pentanol, 1,2-butanediol, 1,2-ethanediol and glycerol in lowering the curvature and/or interfacial tension of the oil-aqueous phases; thus, they form the largest microemulsion regions in the triangular phase diagrams. However, all these non-toxic co-surfactants have good potential for use in preparing palm-based microemulsions for practical applications, such as in pharmaceuticals, personal care products, all-purpose spray liquid cleaners with or without natural insect repellent, microemulsion-spray insecticides for household and animal house use, and as wood preservatives.

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