MICROEMULSIONS FORMED WITH PALM OIL-BASED MATERIALS AS ALL-PURPOSE SPRAY LIQUID CLEANERS FOR HARD SURFACES#

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ABSTRACT

This article describes the formation of microemulsions by utilising fatty acid methyl esters (FAME) and a surfactant or mixed surfactant system derived from palm oil-based oleochemicals. The effects of 1,2 hexanediol as a conventional non-toxic co-surfactant were investigated by observing the ternary phase behaviour of palm oil-based microemulsions for three types of surfactant systems, i.e.*, fatty alcohol ethoxylates 7EO (FAE 7); mixed surfactants FAE 7 and fatty alcohol ethoxylates 2EO (FAE 2) in a 75:25 ratio; and mixed surfactants FAE 7, FAE 2 and methyl ester sulphonate (MES) in a 60:20:20 ratio. The microemulsion (µE) solutions formed were characterised by conductivity, viscosity and droplet-size measurements. The cleaning performance of selected palm oil-based microemulsions as all-purpose spray liquid cleaners for hard surfaces is also reported.*

Keywords: palm fatty acid methyl esters, mixed surfactant system, 1,2-hexanediol, palm-microemulsion spray liquid cleaners.

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INTRODUCTION

Microemulsions are isotropic (clear), have very small droplet size (~8-100 nm), are low in viscosity, and are thermodynamically stable solutions. In such microemulsions, substantial amounts of two immiscible liquids (*i.e.*, water and oil) are brought into a single phase by means of an appropriate surfactant or a mixture of surfactants, with or without a co-surfactant. However, ordinary emulsions appear as milky white dispersions with higher viscosity and droplet size >0.5 µm, and they are only kinetically stable (Hoar and Schulman, 1943; Prince, 1977). Thus, for a number of years, microemulsion systems have attracted the attention of many researchers, formulators, manufacturers and end-users, instigating them to carry out studies and apply these systems in producing high value-added products.

Microemulsions containing low-molecular weight aliphatic or aromatic hydrocarbons have been investigated extensively. However, there is a growing interest in microemulsion systems, which replace hydrocarbon oil with oils derived from natural resources, such as medium alkyl triglycerides and alkyl esters (Alander and Warenheim, 1989; Joubran *et al*., 1993; Hamdan *et al*., 1995; Monig *et al*., 1996; Von Corswant *et al*., 1998; Warisnoicharoen *et al*., 2000; Alany *et al*., 2000; Raman *et al*., 2003; 2005; 2008). Natural base materials have many advantages, such as being renewable, biodegradable, non-flammable, harmless to the environment, and non-toxic to end-users (Leysen, 1992; Hamilton, 1993; Haigh, 1995; Srivastava and Prasad, 2000).

The formation of microemulsions often requires a high concentration of surfactant, but this can sometimes be reduced by using a co-surfactant, *e.g*, 1-pentanol (Prince, 1977; Overbeek *et al*., 1984; Cavalli *et al*., 1996; Raman *et al*., 2003). Kahlweit (1995), Kahlweit *et al.* (1996) and Alany *et al.* (2000)

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found that many short-chain aliphatic alcohols (*e.g.*, 1-pentanol) are regarded as toxic to end-users and aquatic organisms. They also found that 1,2 alkanediols (*e.g.*, 1,2 hexanediol) have similar properties to aliphatic alcohols, although they are found to be less toxic, and, therefore, more suitable as substitutes for use in the formation of microemulsions.

There are numerous applications for microemulsions, as, for example, in detergent formulations (Colgate Palmolive Co., 1987; Azemar, 1997). The most significant property of microemulsions used in detergent formulations is in improving the solubilisation capacity for both polar and non-polar soil compounds. This is due to the very low interfacial tension achieved between the aqueous and oil phases, and thus the spontaneous formation of microdroplets occurs when the components are brought into contact with each other. Other examples of detergent products formulated using the microemulsion system are all-purpose spray liquid cleaners for hard household surfaces (Gasco, 1997; Gross, 2004; Gross *et al.*, 2005; Ismail *et al.*, 2007), all-purpose spray liquid cleaners containing natural insect repellent (Ismail *et al.*, 2008), and other industrial cleaning applications. Therefore, this article discusses the application of palm oil-based derivatives (*e.g.*, palm-based methyl esters, surfactants and mixed surfactants) and conventional non-toxic co-surfactants (*e.g.*, 1,2 hexanediol) in the formation of microemulsions for all-purpose spray liquid cleaners for hard surfaces.

MATERIALS AND METHODS

Materials

Palm fatty acid methyl esters (PFAME) were supplied by Carotech (M) Sdn Bhd, Malaysia. Fatty alcohol ethoxylates 7EO (or FAE 7) and fatty alcohol ethoxylates 2EO (FAE 2) were supplied by Cognis Oleochemicals (M) Sdn Bhd. Palm methyl ester sulphonate (MES) was synthesised in the Advanced Oleochemicals Technology Division (AOTD) of the Malaysian Palm Oil Board (MPOB). The purity of the surfactants ranged from 99.5% to 100% (w/w); however, MES was only ~86% (w/w) pure. The 1,2 hexanediol (or hexylene glycol) and 1-pentanol were bought from Sigma-Aldrich and Fluka Chemical Companies of Singapore and the United Kingdom, respectively.

Methods

The mixtures were prepared by mixing ethoxylated non-ionic and sulphonated anionic surfactants at 10%, 15%, 20%, 25%, 30% and 40% (w/w) with various concentrations of 1,2-hexanediol and 1-pentanol as co-surfactants. Filtered 0.01 M NaCl solution was used as the aqueous phase for preparing the mixtures. The ratio of PFAME to 0.01 M NaCl solution used was 25:75 for both the surfactant FAE 7 and the mixed surfactant FAE 7/FAE 2, but was in the ratio of 30:70 in the mixed surfactant FAE 7/FAE 2/MES system. The mixtures were stirred vigorously, and incubated in a water-bath for ~2 hr at 50±2°C. Then, the mixtures were again stirred vigorously and equilibrated overnight for two days at 25°C (or ambient temperature), and then at 45°C for one month.

The formation of microemulsions and other phases (emulsions or liquid crystals) was observed qualitatively using polarised light sheets. The phases observed were constructed on triangular phase diagrams (Broze, 1997). The microemulsion solutions were characterised for conductivity, viscosity and droplet size. The cleaning performances of selected palm oil-based microemulsions as all-purpose liquid cleaners for hard surfaces were also assessed (Ismail *et al.*, 2007; 2008; Thomas *et al.*, 1996; Japanese Standards Association, 1998).

RESULTS AND DISCUSSION

PFAME were chosen as the oil phase in this study as PFAME have already been shown to have good potential as natural-based oil phases or solvents in agrochemicals (Ismail *et al.*, 2000; 2004), household and industrial cleaners (Choo, 2004a, b; Aishah, 2004; Yeong, 2006), lubricants (Choo, 1995; Loh, 2006) and other household and industrial applications. The study also focused on the concentrations of the surfactants, in the range of 10% to 40% (w/w) that can be applied in the preparation of the microemulsions for practical applications.

Partial Ternary Phase Diagrams of PFAME/Water/Surfactant or Mixed Surfactants/Co-surfactants

Partial ternary phase diagrams show that the minimum concentration of the surfactant FAE 7 (*Figure 1*), mixed surfactant FAE 7/FAE 2 at the 75:25 ratio (*Figure 2*), and mixed FAE 7/FAE 2/MES at the 60:20:20 ratio (*Figure 3*) for forming microemulsions was 10% (w/w). However, the concentrations of 1,2-hexanediol required were approximately 30%, 20% and 40% (w/w) to form microemulsions with 10% (w/w) of FAE 7, mixed FAE 7/FAE 2 and mixed FAE 7/FAE 2/MES solutions, respectively, at room temperature.

The optimum concentrations for both FAE 7/1,2-hexanediol and mixed FAE 7/FAE 2/MES/1,2-hexanediol for forming microemulsions were 20% and 15% (w/w), respectively, at both the

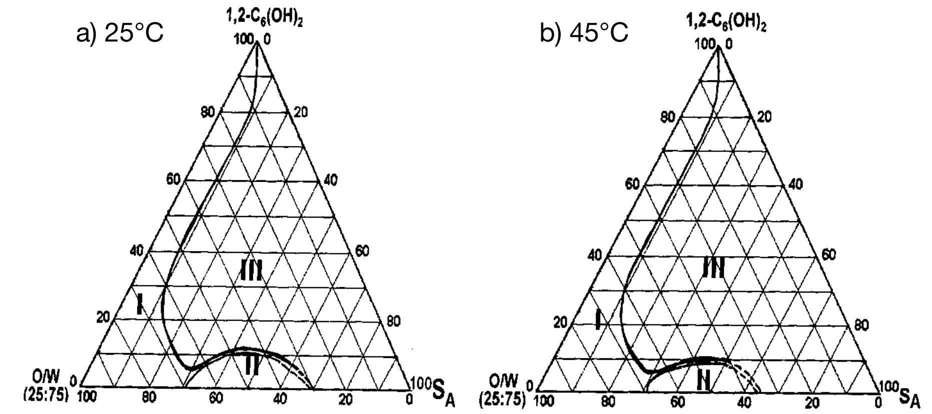

Figure 1. Effects of 1,2-hexanediol [or 1,2-$C_6(OH)_2$] on partial ternary phase diagram of FAE 7 (or S_A) solutions and 25:75 of PFAME/H_2O at (a) 25°C and (b) 45°C. The I, II, and III are the two/multiple-phases (or emulsions), liquid crystals, and oil/water and/or water/oil microemulsions. S and O/W refer to surfactant and ratio of oil to water, respectively.

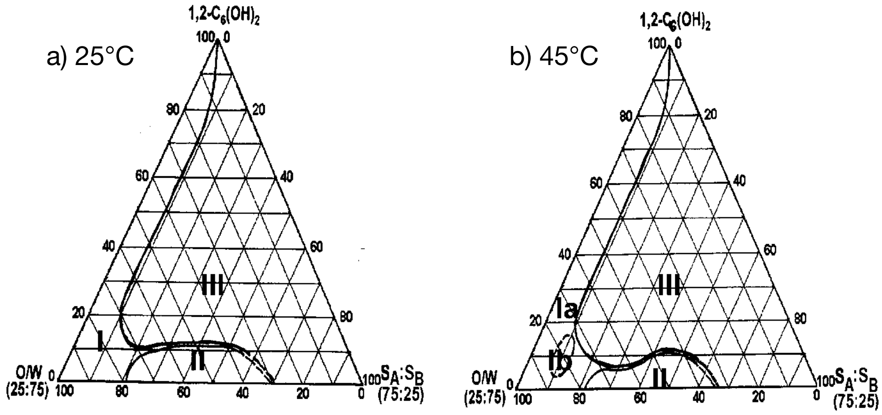

Figure 2. Effects of 1,2-hexanediol [or 1,2-$C_6(OH)_2$] on the partial ternary phase diagram of 75:25 of FAE 7/FAE 2 (or S_A/S_B) and 25:75 of PFAME/H_2O at a) 25°C and b) 45°C. The I, II and, III are two/multiple-phase (or emulsions), liquid crystals, and oil/water and/or water/oil microemulsions. S and O/W refer to surfactant and ratio of oil to water, respectively.

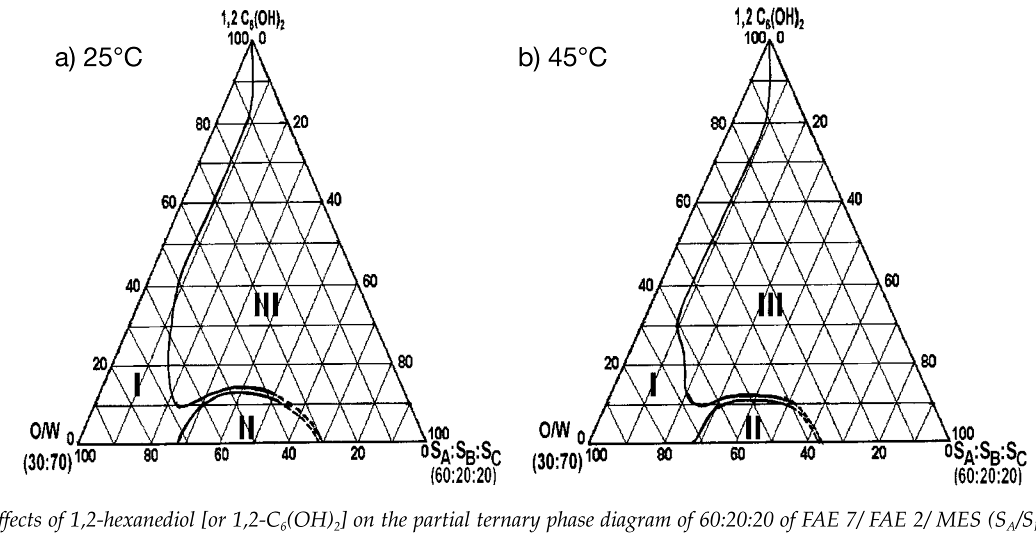

Figure 3. Effects of 1,2-hexanediol [or 1,2-$C_6(OH)_2$] on the partial ternary phase diagram of 60:20:20 of FAE 7/ FAE 2/ MES (S_A/S_B/S_C) and 30:70 of PFAME/H_2O at a) 25°C and b) 45°C. I, II and, III are two/multiple-phase (or emulsions), liquid crystals, and oil/water and/or water/oil microemulsions. S and O/W refer to surfactant and ratio of oil to water, respectively.

temperatures of 25°C and 45°C (*Figures 1* and *3*). However, the optimum concentration for mixed FAE 7/FAE 2 and 1,2-hexanediol was 20% and 10% (w/w), respectively, at both 25°C and 45°C (*Figure 2*). In addition, there was a three-phase region observed in the PFAME/mixed FAE 7/ FAE 2/1,2 hexanediol/water mixture at 45°C (*Figure 2b*). This three-phase region consisted of a bicontinuous (or middle-phase) microemulsion containing comparable amounts of water and oil phases coexisting with both an excess of oil and water.

These results indicate that the solubility of PFAME and water at a 25:75 ratio of oil to water in mixed FAE 7/FAE 2 solution was slightly higher than in FAE 7 and mixed FAE 7/FAE 2/MES solutions when 1,2-hexanediol was added as the co-surfactant. This means that the mixed FAE 7/FAE 2 solution had a slightly higher capacity to solubilise PFAME and water than the FAE 7 and mixed FAE 7/FAE 2/MES solutions, thus leading to a bigger region of microemulsion solutions being formed.

The effect of 1,2-hexanediol as a non-toxic co-surfactant on the formation of PFAME-microemulsions was compared to the effect of 1-pentanol, the medium chain aliphatic alcohol (*Figure 4*) (Raman *et al.,* 2005; Ismail, 2006). The microemulsion regions formed with the PFAME/ surfactants (FAE 7 or mixed FAE 7/FAE 2 or mixed FAE 7/FAE 2/MES)/1,2 hexanediol/water) (*Figures 1, 2* and *3*) were larger than that of the PFAME/surfactant FAE 7/1-pentanol/ water system (*Figure 4*).

This shows that FAE 7, mixed FAE 7/FAE 2 (at a ratio of 75:25), and mixed FAE 7/FAE 2/MES (at a ratio of 60:20:20) with addition of 1,2-hexanediol had higher solubilisation capacity for both PFAME and water than 1-pentanol as the co-surfactant. The results also indicate that 1,2-hexanediol showed a better synergistic effect as a co-surfactant than 1-pentanol to single or mixed non-ionic surfactant systems in lowering the interfacial tension and/or the curvature between the oil and aqueous phases. Thus, larger microemulsion regions with very small droplets were formed.

Cleaning Performance of Palm Oil-based Microemulsions (µE) as All-purpose Liquid Cleaners

Cleaning tests were conducted with palm oil-based microemulsion liquid cleaners (*Figure 5*) using a detergent-cleaning tester 10 (DCT 10) machine (*Figure 6*). This machine, also called a wet

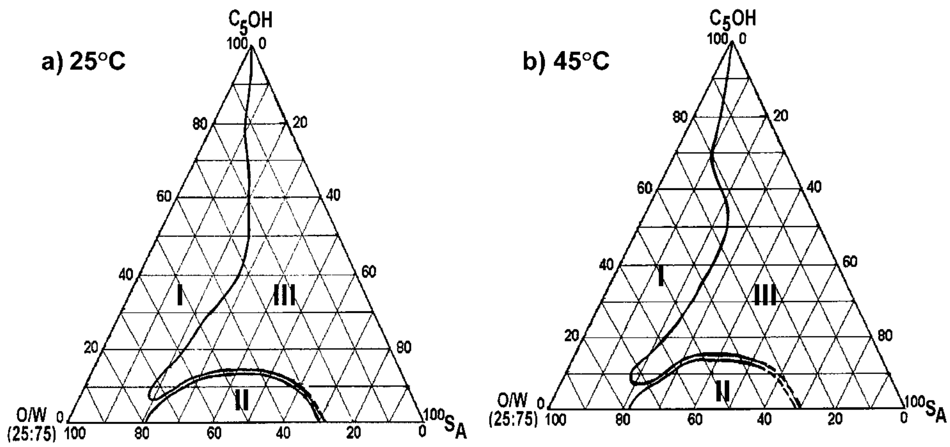

Figure 4. Effects of 1-pentanol as co-surfactant on FAE 7/PFAME/water system at (a) 25°C and (b) 45°C. O/W and S refer to oil to water ratio and surfactant, respectively. O/W ratio was 25:75, and I, II and III represent the emulsion (two/three-phase), liquid crystalline (LC) and microemulsion regions, respectively.

Figure 5. Palm-µE liquid cleaners.

Figure 6. Detergent cleaning tester 10 (DCT 10).

abrasion scrub tester or double-headed washability tester, is designed to provide an accelerated method for determining abrasive wear resistance of surface coatings/materials, and also to test the performance and abrasive effect of cleaning compounds. The methods to evaluate cleaning performance on hard household surfaces have been described in the past (Thomas *et al*., 1996; Japanese Standards Association 1998).

The formulated cleaning products were evaluated for their capacity to clean off soil on tiles (or hard surfaces). Cleaning performance was measured using a reflectometer. The reflectometer, also known as a gloss meter, is used to determine or measure the reflectance of light from a surface. This instrument is used extensively in various fields, from paint to auto manufacture, to measure the amount of reflected light or glossiness of a surface. Together with the detergent-cleaning tester, the gloss meter is used to measure the glossiness of hard surfaces before and after the cleaning test. The most commonly used model is the Glossmaster 60°, which can be bought from Sheen Instrument Ltd, England. This gloss meter is supplied with a cleaning tester in accordance to ASTM, ISO and DIN Standards.

Three optimum compositions of palm-microemulsion liquid cleaners were chosen from the partial ternary phase behaviour studies as discussed above, *i.e.* F1, which consisted of FAE 7 only; F2, which consisted of mixed FAE 7/FAE 2 at a 75:25 ratio; and F3, which consisted of mixed FAE 7/FAE 2/MES at a 60:20:20 ratio. The concentrations of the surfactant (or mixed surfactants) used were 15% and 20% (w/w), respectively, and that of 1,2 hexanediol (or co-surfactant) used was 20% (w/w). Only the ratio of 25:75 of PFAME to water was chosen because the study emphasises a system with a large amount of water (or aqueous phase).

The physical properties of the palm-microemulsions (μE) as an all-purpose liquid cleaners for hard surfaces are shown in *Table 1*. The particle size, viscosity and pH values were approximately equivalent in all the formulations. However, the conductivity values varied from 109 to 919 µSm and from 136 to 976 µSm for 15% and 20% (w/w) of surfactant, respectively. With their very small droplet size (~7.5 to 9.5 nm) and low viscosity, the solutions can be applied onto hard surfaces in a diluted or concentrated form. Thus, we have demonstrated that the palm-microemulsions as liquid cleaning formulations have potential to be superior grease and oily soil removers compared to powdered cleaning formulations (Gross, 2004; Gross *et al*., 2005). As described previously, the most significant detergency properties of microemulsions are their improved solubilisation capacity for both polar and non-polar soil compounds, their very low values of interfacial tension between aqueous and oil phases, and the spontaneous emulsification when the components are brought into contact with one another (Azemar, 1997; Salager, 1999).

Cleaning performance was tested on glazed white tiles (as typical hard surfaces) using a detergent cleaning tester (DCT) machine. The microemulsion solutions were diluted at ratios of 1:0; 1:1; 1:3; 1:5 and 1:9 of palm-microemulsion liquid cleaner to water. The results show that all the formulae tested had good performance as cleaning solutions with 15% and 20% (w/w) concentrations of surfactant or mixed surfactants even after dilution up to a ratio of 1:9 as shown in *Figures 7* and *8*, respectively. Furthermore, the palm-microemulsion solutions formed with the mixed surfactants FAE 7/FAE 2/MES gave better cleaning performance than the solutions formed with the mixed surfactants FAE 7/FAE 2 and with surfactant FAE 7 at both 15% and 20% (w/w) concentrations (*Figures 7* and *8*).

These results indicate that the mixed non-ionic and anionic surfactants have better synergistic effects than mixed non-ionic surfactants or single non-ionic surfactant when the palm-microemulsions were used as liquid cleaners. It was also found that the palm-microemulsion liquid cleaners showed superior cleaning performance, and gave better and a longer-lasting shiny appearance to the cleaned surface than a conventional liquid cleaner. The addition of a natural insect repellent (N.I.R) to the palm-microemulsion liquid cleaners enhanced value-added to these all-purpose liquid cleaners as well as enhanced the importance of such products to public health (HAPPI, 2006). Ismail *et*

TABLE 1. PROPERTIES OF PALM-MICROEMULSION (µE) LIQUID CLEANERS

Formula	Conductivity (µSm)	Particle size (nm)	Viscosity (cP)	pH
F1: 15% (FAE 7)	109	8.8	13.1	5.0
F2: 15% (FAE7/FAE 2)	137	9.5	12.6	5.0
F3: 15% (FAE 7/FAE 2/MES)	919	8.8	12.7	4.8
F1: 20% (FAE 7)	136	7.9	17.0	5.2
F2: 20% (FAE7 + FAE 2)	141	9.0	15.4	5.4
F3: 20% (FAE 7 + FAE 2 + MES)	976	7.5	15.1	5.1

Note: FAE 7 and FAE 2 are ethoxylated non-ionic surfactants, and MES is a sulphonated anionic surfactant.

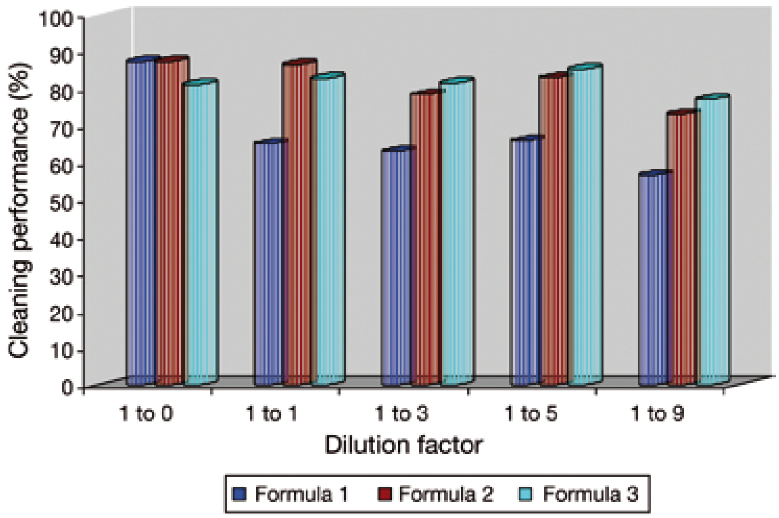

Figure 7. Cleaning performance of palm-microemulsion liquid cleaners at 15% concentration of surfactant or mixed surfactants and at various ratios of dilution. 1 to 0, 1 to 1, 1 to 3, 1 to 5 and 1 to 9 were the dilution ratios.

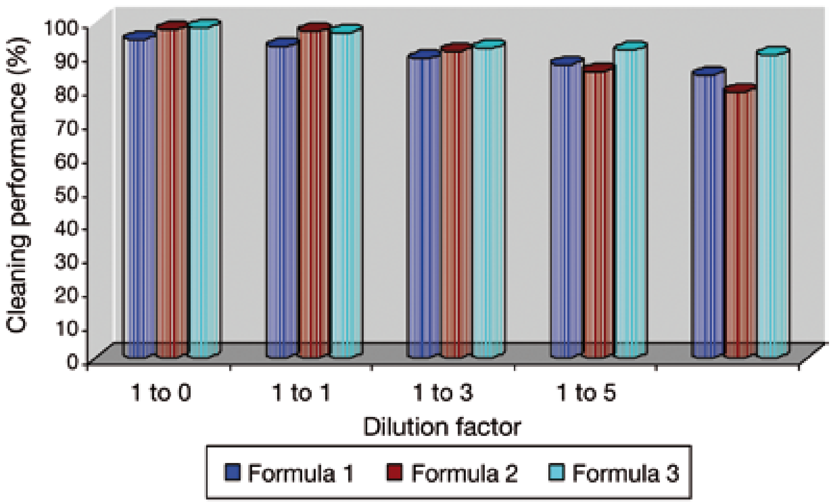

Figure 8. Cleaning performance of palm-microemulsion liquid cleaners at 20% concentration of surfactant or mixed surfactants, and at various ratios of dilution. 1 to 0, 1 to 1, 1 to 3, 1 to 5 and 1 to 9 were the dilution ratios.

al. (2007; 2008) confirmed that the addition of 5% and 7% (w/w) of a local N.I.R (*e.g.* citronella oil) to palm-microemulsion liquid cleaners showed significant effects in repelling mosquitoes which were comparable to conventional insect repellents (diethyl-3-methylbenzamide or DEET) at 10% (w/w).

CONCLUSION

The palm-microemulsion liquid cleaners formulated are 2-in-1 products, *i.e.* they clean oil- or water-soluble soils, and they repel insects on hard surfaces. They are superior grease and oily soil removers compared to powdered cleaning formulations. Furthermore, palm-microemulsion liquid cleaners are terpene-free, have good cleaning performance and impart a longer-lasting shiny effect to the cleaned surface than conventional liquid cleaners. Thus, the products are a viable option to use for cleaning hard surfaces. The palm-microemulsions as all-purpose liquid cleaners can be supplemented with oil-soluble active ingredients, such as natural insect repellents, disinfectants and fragrances, which can value-add to the products.

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