

Microemulsion-based palm kernel oil extraction using mixed surfactant solutions

Apasee Naksuk^{a,d}, David A. Sabatini^b, Chantra Tongcumpou^{a,c,*}

^a National Center of Excellence for Environmental and Hazardous Waste Management, Chulalongkorn University, Phatumwan, Bangkok 10330, Thailand

^b Civil Engineering and Environmental Science Department, Carson Engineering Center, 202 W. Boyd, University of Oklahoma, Norman, USA

^c Environmental Research Institute, Chulalongkorn University, Pathumwan, Bangkok 10330, Thailand

^d International Postgraduate Programs in Environmental Management, Graduate School, Chulalongkorn University, Pathumwan, Bangkok 10330, Thailand

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ABSTRACT

This study introduces a novel technique using surfactant microemulsion-based oil seed extraction. To achieve this objective, microemulsion formation with palm kernel oil was studied first. Then, the selected microemulsion system was used for palm kernel extraction. The results showed that the mixed surfactant of 3 wt% Comperlan KD and either 0.1 wt% Alfoterra145-5PO or 145-8PO provided an ultralow interfacial tension with the palm kernel oil (0.0197 and 0.0359 mN/m, respectively). By using those two aqueous surfactant systems for palm kernel oil extraction, the extraction efficiency was 93.99 and 94.13% at the optimum crushed kernel size between 0.212 and 0.425 mm, using 1 g seed load to 10 ml of solution and 30 min of contact time. The extracted oil quality was evaluated for water content, fatty acids composition and surfactant partitioning into oil phase. The results showed that the quality of the oil obtained using the surfactant microemulsion-based technique is of similar or better quality than when extracted by hexane solvent.

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1. Introduction

Due to the energy crisis, several oil-bearing crops have become of interest for use as a raw material for biofuel. While there are more than 350 types of oil-bearing crops, the ones that have been commercialized to date include soybean (*Glycine max*), rapeseed (*Brassica napus*), sunflower (*Helianthus annuus*), and oil palm (*Elaeis guineensis*) (Ayhan, 2005). However, to be useful either for food or energy, first the oil must be extracted from the plants' seeds. Vegetable oil can be extracted by several methods including mechanical and solvent extraction. Solvent extraction is a conventional method that is widely used at a large-scale production for obtaining vegetable oils from their seeds, i.e. peanut (*Arachis hypogaea*), soybean, sunflower, corn (*Zea mays*) and palm kernel (Mattil et al., 1964). However, hexane extraction, the most common type of solvent extraction, requires expensive equipment to handle the solvent and to ensure worker safety measure because hexane is a highly volatile solvent. Hexane is classified as a hazardous air pollutant by the US Environmental Protection Agency and they thus consider vegetable oil extraction plants to represent

a potential major source. It is estimated that 0.7 kg of hexane per ton of seed is released into the environment (United State Environment Protection Agency, 2005). Exposure to hexane at 125 ppm for 3 months causes peripheral nerve damage, muscle wasting, and atrophy (Agilent Technology, MSDS, Agilent Technology, 2008). For this reason, alternative solvent extraction methods are needed that eliminate the use of toxic compounds such as hexane (Tyson et al., 2004). Several researchers have studied aqueous-based extraction (Hagenmaier, 1974; Southwell and Harris, 1992; Evon et al., 2007; Wu et al., 2009). From Wu et al. (2009), using enzyme-assisted aqueous extraction to demulsify of oil-rich emulsion increased their extraction efficiency for soybean yield free oil to 88% from the total 90% oil available.

In this study microemulsion-based extraction was evaluated as an alternative to hexane for vegetable oil extraction. This approach is considered a clean technology since surfactants used for the extraction are non-toxic substances and biodegradable. This technique is based on microemulsion formation in which a surfactant plays a key role in the process by lowering interfacial tension between the aqueous extracting system and the oil vegetable seeds. As a result, oil can be extracted from the seed meals.

Several research studies have evaluated the possibility of microemulsion formation using vegetable oil and various types of surfactant. Raman et al. (2003) studied microemulsion formation by mixture of non-ionic surfactant with palm oil and its derivatives. Vegetable oil containing triglycerides as the major com-

* Corresponding author at: Environmental Research Institute, Chulalongkorn University, Pathumwan, Bangkok 10330, Thailand. Tel.: +66 2 2188138; fax: +66 2 2188210.

E-mail address: tchantra@chula.ac.th (C. Tongcumpou).

ponents is one of the most difficulty oils to form a microemulsion with. Selecting suitable surfactant system is the first important step for microemulsion formation with vegetable oil. Recently, our group has evaluated the phase behavior of vegetable oil in microemulsion systems using lipophilic linker and extended surfactants (Komesvarakul et al., 2006; Do et al., 2009). The behavior of soybean oil in a water emulsion stabilized by non-ionic surfactant also was reported by Hsu and Nacu (2003). Microemulsion formation of eucalyptus oil by mixing nonionic surfactant (Brij 35) and anionic surfactant (AOT) using butanol alcohol as linker was reported by Rajib and Bidyut (2005). More closely to this present work, in our working group, Klongkleaw (2005) was able to extract soybean oil using non-ionic surfactant (Comperlan KD) solution. A similar system was developed in order to extract oil from palm kernel seed. Thus, while researchers have studied surfactant interactions with vegetable oil, to date no one has published work on microemulsion-based extraction on palm kernel seeds as an alternative to hexane extraction.

In Thailand and South-east Asian countries, palm oil is a major oil-bearing crop due to the tremendous growth of the oil palm industry in the region. Palm oil has good potential to be developed as an energy crop. Palm oil accounts for 70% of the Thai vegetable oil market, and was estimated to be worth 40,000 million Baht per annum (Chavalparit et al., 2006). In 2003, the production of palm oil was estimated to be 680,000 tons and increased to 718,000 tons in 2006.

Thus, the main objective of this work was to investigate the possibility of using the surfactant microemulsion-based process for oil extraction from palm kernel seeds. The surfactant solution used in this study was a solution of non-ionic and anionic extended surfactants. The anionic extended surfactant used in this study has been shown to lower the interfacial tension (IFT) between vegetable oil and aqueous surfactant solution down to 10^{-3} mN/m or so-called ultralow IFT (Witthayapanyanon et al., 2006). The optimum conditions for extraction were investigated based on extracted oil yield. In addition, the quality of extracted oil between this proposed method and conventional method for extraction also were compared.

2. Experimental

2.1. Materials

The two anionic extended surfactants evaluated were sodium alkyl polypropylene oxide sulfate ($R-(PO)_x-SO_4Na$ with alkyl(R) group consisting of a branched hydrocarbon chain with 14–15 carbons (C_{14-15}) and with five propylene oxide units – known as Alfoterra145-5PO – which was 28.7% active or eight propylene oxide units – known as Alfoterra145-8PO – which was 29.1% active. These surfactants were donated from SaSol North America Inc. The non-ionic surfactant evaluated was coconut fatty acid diethanolamine (Comperlan KD) which was 98% active. This surfactant was purchased from Cognis, Thailand. The palm kernel oil and palm kernel seed used in this work were donated from Lamsoong Thailand Company and used as received during July to December 2006.

2.2. Methods

To select the surfactant system to use for extraction, preliminary experiments were conducted to find systems capable of forming microemulsions with palm kernel oil. Next, phase behavior studies of the selected surfactant system were conducted by varying the system salinity in order to obtain the system that provided the lowest IFT. The phase behavior scan was done following the method described in Tongcumpou et al. (2003a). These selected systems

from the phase study were subsequently used for extracting initial oil from palm kernel seeds. Efficiency of oil extraction was determined against the average amount of oil content in palm kernel seeds at 48–59 wt% (Gunstone, 2002). Parameters to determine the optimum conditions for the extraction were contact time and palm kernel load. All extraction experiments were done in triplicate.

IFT values in the phase behavior study were measured by a spinning drop video tensiometer (Dataphysics, SVT 20 Model) following the method described in Acosta et al. (2004). Fatty acids composition was measured using GC-FID by derivatizing fatty acids into methyl esters in January 2007. Water content in oil phase was measured by Karl fisher titration. The surfactant concentration remaining in water phase was determined by HPLC-ELSD and by titration method ASTM D1681-92 for Comperlan KD, and for Alfoterra145-5PO and 145-8PO, respectively.

3. Results and discussion

3.1. Phase behavior of palm oil and relation to oil extraction

From preliminary studies, the mixed surfactant system of both extended anionic Alfoterra145-5PO and Alfoterra145-8PO and nonionic Comperlan KD were able to form microemulsions with palm kernel oil at the concentration (wt%) ratio of anionic to non-ionic equal 0.1–3.0. Therefore, two systems were selected; System A (Mix 3% Comperlan KD and 0.1% Alfoterra145-5PO) and System B (Mix 3% Comperlan KD and 0.1% Alfoterra145-8PO) were scanned with different NaCl in the range of 0–20 wt%. The interfacial tension was then measured to evaluate phase transition of the two systems. The results (Fig. 1) show that both extended anionic surfactants mixed with the non-ionic surfactant in salinity scans systems generated microemulsion phase transition. The IFT values of systems with different salt concentrations varied from 1 to 10^{-3} mN/m. IFT values in the range of 10^{-2} to 10^{-3} mN/m are considered ultralow and desirable. Thus, microemulsion formation

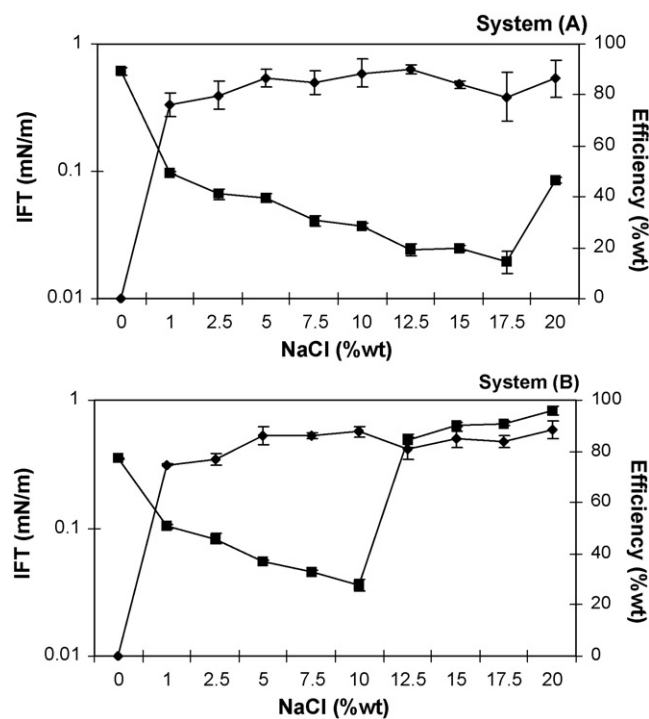


Fig. 1. The relationship between IFT (mN/m) and extraction efficiency at grain size 0.212–0.425 mm with System A (Mix 3% Comperlan KD and 0.1% Alfoterra145-5PO) and System B (Mix 3% Comperlan KD and 0.1% Alfoterra145-8PO) at different NaCl concentrations.

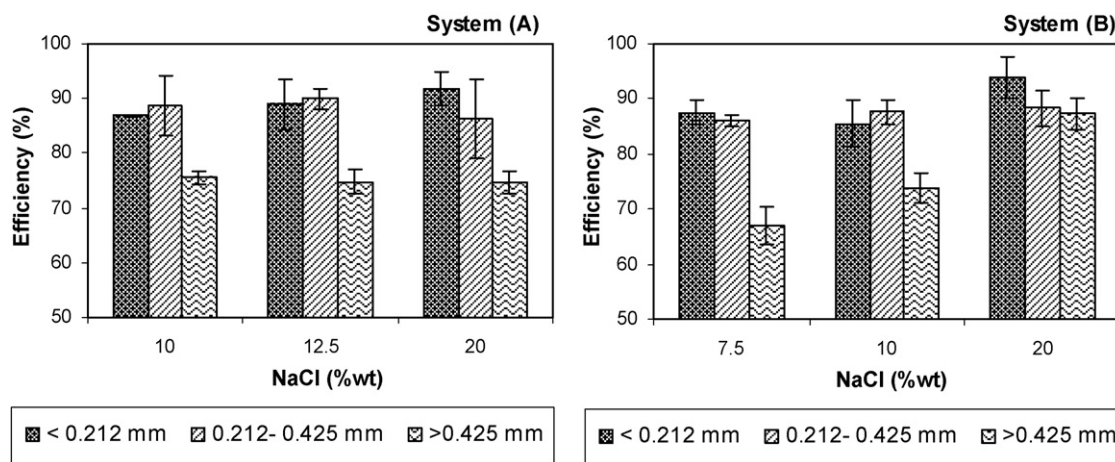


Fig. 2. The effect of grain size of ground seeds on the palm kernel oil extraction efficiency (wt%) of the mixed surfactant solution; System A (Mix 3% Comperlan KD and 0.1% Alfoterra145-5PO) and System B (Mix 3% Comperlan KD and 0.1% Alfoterra145-8PO) at different NaCl concentrations.

using these two systems of mixed surfactants looks promising for palm kernel oil extraction. As expected the IFT values decreased as the salinity increased because adding salt promoted the formation of middle phase microemulsion due to the reduction in repulsive forces between the ionic charges of surfactants at their head group (Tongcumpou et al., 2003a). Thus, an increase in salinity enhanced the phase transition of Winsor type I microemulsion toward Winsor type III microemulsion.

These two mixed surfactant systems – System A and System B – with different salinities also were used for palm kernel oil extraction using a ratio of 1 g ground palm kernel seed to 10 ml of the mixed surfactant solutions, and 30 min contact time. The efficiency of oil extraction was plotted related to IFT values from the phase behavior (Fig. 1). It can be seen that with NaCl addition to the system IFT reduced and hence oil seed extraction was enhanced. However, IFT may not be linearly correlated to oil extraction. This indicates that IFT was not the only parameter governing the oil extraction efficiency but other parameters also played a role in oil extraction. These results are similar to the studies by Tongcumpou et al. (2003b, 2006) on removal of motor oil and palm oil from fabric. They found that while high efficiency of oil removal from fabric resulted for the lower IFT values, other parameters (i.e. oil solubilization and coalescence rate) also appeared to impact the overall oil removal (or extraction) efficiency. These encouraging results on microemulsion-based oil seed extraction motivated a preliminary investigation on parameters that influenced the optimum efficiency of the oil extraction and that will be described in the next aspects.

In this work we used a surfactant concentration of around 3 wt% to ascertain that the microemulsion was dominant in the system—future work will explore lower surfactant concentration approaching the critical microemulsion concentration (cmc).

3.2. Palm kernel oil extraction

As mentioned above, other parameters that could impact efficiency of oil extraction include grain size, contact time and palm kernel loading. However, since salinity did not exhibit a significant difference there were three salinities selected for each surfactant systems; 10, 12.5 and 20%; and 7.5, 10 and 20% NaCl (wt%) for System A and System B, respectively. It should be noted here that high salt was needed to reduce IFT of the microemulsion system and hence to extract oil from seeds. However, most of the salt is dissolved in aqueous solution and had little effect on extracted oil as well as residual meal. Nonetheless, future systems can be designed to lower the salt requirement as needed (Elliott et al., 2004).

3.2.1. Effect of grain size

The palm kernel seeds were varied in three sizes following the standard of the vegetable oil seed size of US. There were coarse size (larger than 0.425 mm), a fine size (between 0.212 and 0.425 mm) and a very fine size (less than 0.212 mm). The experiment was conducted by adding 10 ml of the mixed surfactant system into the tube and homogenized with 1 g of ground palm kernel. The results showed that both selected surfactant systems were able to extract palm kernel oil from palm kernel seed. Since the grain size was a physical property, it was expected that the effect of grain size would follow the same trend for both surfactant systems, hence for this experiment, only System A was evaluated. It was found that the largest grain size showed the lowest efficiency while the other two sizes showed similar extraction efficiencies (Fig. 2). This suggested that the smaller size of seeds provide larger surface area enhancing the capability of the surfactant monomer to interact with the surface and reduce interfacial tension thereby liberating oil from the seeds. Hence, the middle size range (0.212–0.425 mm) was the most appropriate grain size since the smaller size would require more energy for its grinding process. As a consequence, for the next-step the impact of contact time, the 0.212–0.425 mm grain size of ground seeds was evaluated.

3.2.2. Effect of contact time

The contact time indirectly indicates the rate of reactions on detachment of oil from solid seeds. However, since for this oil extraction process, solubilization of the oil into micelles is not desirable, the optimum contact time should be examined. In this experiment, the contact times were varied from 15, 30, 45 and 60 min. The results show similar trend for both Systems A and System B that the contact times up to 30 min had the maximum efficiency, and then extraction efficiency after 30 min tends to be more or less decreased (Fig. 3). Thus, for the two systems, the contact time at 30 min was considered as the optimum time for extraction. Thus, 30 min was enough time for reducing IFT of oil and solid surface and thus let oil to detach in the aqueous surfactant solution. However, for longer contact times free oil solubilized more into the micelles resulting in less free oil.

3.2.3. Effect of palm kernel load

In this experiment, the kernel loads (solid to liquid ratio) were 0.5, 1.0, 1.5 and 2.0 g with 10 ml of mixed surfactant solution, 30 min contact time. Fig. 4 shows that the optimum of extraction efficiency appeared at 1 g oil seeds loading for all three salinities in both systems. When the palm kernel loaded into the system was

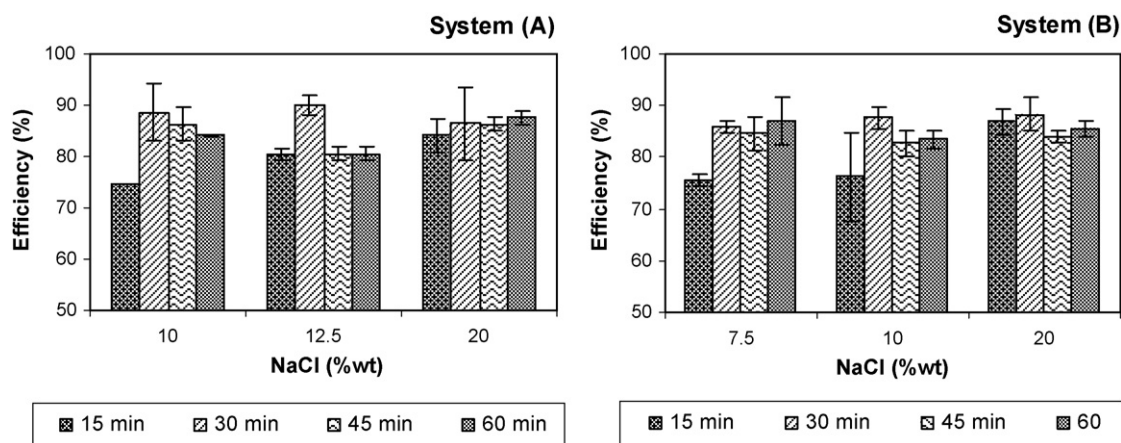


Fig. 3. The effect of contact time on the palm kernel oil extraction efficiency (wt%) of the mixed surfactant solution; System A (Mix 3% Comperlan KD and 0.1% Alfoterra145-5PO) and System B (Mix 3% Comperlan KD and 0.1% Alfoterra145-8PO) at different NaCl concentrations.

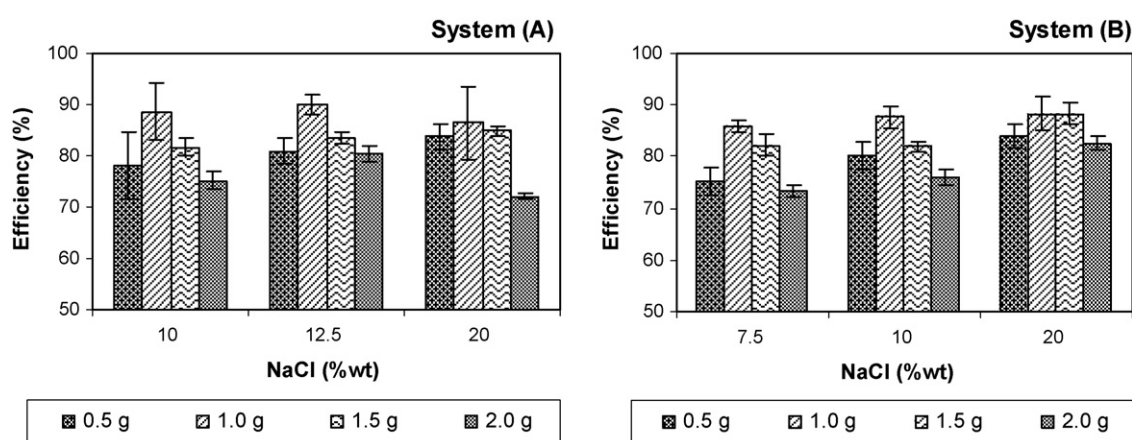


Fig. 4. The effect of kernel loading (solid to liquid ratio—volume = 10 ml) on the oil extraction efficiency (wt%) of the mixed surfactant solution; System A (Mix 3% Comperlan KD and 0.1% Alfoterra145-5PO) and System B (Mix 3% Comperlan KD and 0.1% Alfoterra145-8PO) at different NaCl concentrations.

increased, the extraction efficiency tended to decrease because a higher mass of palm kernel load led to less penetration of surfactant monomers into the kernel and less coalesce between surfactant monomers and oil.

At this point, from the results on the effects of salinity, grain size, contact time and loading the optimum condition for oil extraction for both systems were 10% NaCl, ground seed size 0.212–0.425 mm, contact time of 30 min and 1.0 g loading per 10 ml of the surfactant solution. These conditions will be used to compare the yield and scale up to produce oil for oil quality analysis to compare with the oil extracted by hexane using Soxlet method.

3.3. Compare efficiency with hexane extraction

Palm kernel oil extraction by hexane also was carried out using the same ground seed and same load of seeds to compare the efficiency to our optimum systems; grain size was 35–65 mesh, 30 min of contact time and 1 g of palm kernel loading. The extraction experiments were done using a reflux Soxlet method. Fig. 5 compares the extraction efficiency of palm kernel oil using hexane and our surfactant systems: System A and System B. The results showed that the efficiencies of oil extraction from both the microemulsion-based of System A and of System B were statistically the same as the hexane extraction. In addition to extraction yield, in order to induce the microemulsion-based extraction to replace hexane extraction, the extracted oil quality was examined to evaluate whether it was similar to the one obtained from hexane extraction, as discussed in the next section.

3.4. Oil quality

The parameters selected for the quality of extracted oil were the water content of the extracted oil, the amount of surfactant penetration in oil phase and the fatty acids composition in extracted oil. In addition, the oil obtained from hexane extraction was evaluated for comparison. The results are given in Table 1. From visual observation, the appearance such as color and clearness of extracted oil from surfactant aqueous-based systems were very similar to the

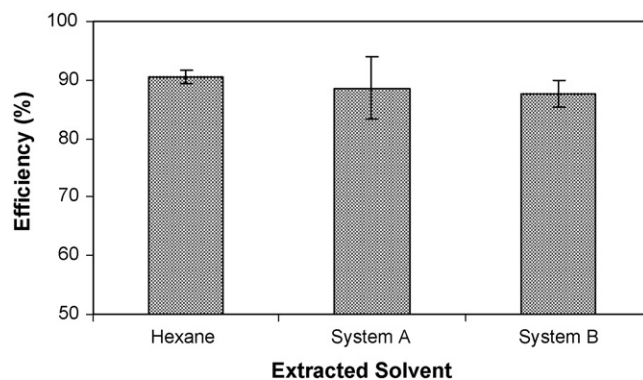


Fig. 5. Comparison of the extraction efficiency for palm kernel oil using hexane extraction method and surfactant aqueous-based method; System A (Mix 0.1% Alfoterra145-5PO and 3% Comperlan KD at 10% NaCl) and System B (Mix 0.1% Alfoterra145-8PO and 3% Comperlan KD at 10% NaCl).

Table 1

The comparison of oil quality between hexane extraction and microemulsion-based system; System A (Mix 3% Comperlan KD and 0.1% Alfoterra145-5PO) and System B (Mix 3% KD and 0.1% Alfoterra145-8PO) with 10% NaCl.

Parameters	Extraction methods		
	Hexane	Surfactant aqueous-based systems	
		System A	System B
Color	Clear yellow	Clear yellow	Clear yellow
Water in oil (wt%)	0.385	0.191	0.223
Surfactant remaining in water phase (wt%)			
Comperlan KD	–	0.602 ± 0.041	0.03
AF 145-5PO	–	0.099 ± 0.3640	–
AF 145-8PO	–	–	0.090 ± 0.003
Fatty acid (wt%)			
C12	49.41	49.52	49.64
C14	17.56	17.44	17.47
C16	9.24	9.17	9.15
C18:0	2.73	2.74	2.74
C18:1	18.18	18.21	18.05
C18:2	2.88	2.92	2.90

one obtained from the Soxlet hexane extraction. Water content in the oil extracted with surfactants met the standard value of good oil quality that should not exceed 0.5 wt% (Gunstone, 2002) and it was found even lower than the one in the oil extracted by hexane. Fatty acids compositions for all extracted solvent were not significantly different.

The amount of Comperlan KD remaining in the aqueous phase was reduced which means this non-ionic surfactant partitioned into the oil phase while extended ionic surfactant concentration was found relatively high in the water phase. This may be explained by the fact that Comperlan KD was a non-ionic surfactant produced from coconut oil which had similar composition of fatty acids found in palm kernel oil. Thus, the non-ionic surfactant may be soluble in the oil phase and some may adsorb on the residual meal. Nonetheless, since this non-ionic surfactant is considered as edible surfactant, this will not affect the quality of extracted oil or the meal if it is used for consumer products/feedstocks, only the cost of the surfactant that must be replaced as the extraction is recycled needs to be considered. Further information revealed that the non-ionic surfactant Comperlan KD used in this research produced by Cognis Company which claimed their product had an eco-label as “Good environmental choices” by the Swedish Society for Natural Conservation. Therefore, although this surfactant exists in oil phase, it can be considered safer than hexane.

4. Conclusions

Microemulsion formation using an aqueous-based surfactant system offers several advantages. First, it can reduce toxic substance exposed to the environment due to the absence of organic solvents for extraction process. Second, low energy requirements for total process since the microemulsion of mixed surfactant can be produced at room temperature and quite insensitive to temperature. Finally, it is worth considering from both of economic and environmental points of views because of lower energy consumption, uses non-toxic chemicals in the process, and less pollution emission and waste generation. In other words, it may be concluded that the approach of this present work introduced an alternative technique considered a green chemistry or clean technology. Conclusively speaking, the aqueous microemulsion-based system can be considered as a promising alternative approach for oil extraction industry in the future.

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