

Mixed Aqueous Surfactants Solution for Rapid Oil Extraction from Palm Kernel Seed

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ABSTRACT

Adverse effects associated with the use of hexane or other similar organic extraction solvents for extracting oil from oilseeds have been a drive for alternative less toxic oil extracting solvents. This research focuses on oil extraction from grinded palm kernel using solution of mixed aqueous polyoxyethylene (23) lauryl ether (BRIJ-35) and sodium dodecyl sulphate (SDS) surfactants and sodium chloride (NaCl) as the electrolyte. Grinded Biomass was agitated in the solution of the mixed surfactant, allowing the extracted oil to be liberated from the seeds as separate phase from the aqueous phase. The effect of SDS concentration, NaCl concentration, contact time and liquid/seed ratio on oil yield was studied. The extracted oil was subjected to Fourier Transform Infrared Spectroscopy (FT-IR) characterization to determine the functional groups present in the oil. At optimum conditions for the mixed aqueous surfactant extraction (0.2379 wt.% SDS, 0.5995 wt.% BRIJ-35, 0.3wt.% NaCl, 10 ml/g liquid/solid ratio, 25 min contact time at 25°C), an optimum 78% oil yield was obtained. In contrast, at 50°C for 24hr, an optimum of 81% and 82% oil yield were obtained respectively when hexane and petroleum ether were used as oil extracting solvents. Addition of BRIJ-35 and NaCl to the SDS solution improved the oil yield. Oil extracted using mixed aqueous surfactants, petroleum ether and hexane have the same functional groups indicating the surfactant does not affect the quality of the oil extracted.

Keywords: Surfactant; hexane; petroleum ether; palm kernel; extraction

Introduction

Fuels from fossil sources are irreversibly depleting and combustion from this fuel source has negative environmental effect. Prevention of environmental pollution and energy crisis associated with the use of this fuel source as complete source of energy has been a major drive for the search for sustainable oil extraction from

oil-seeds for biofuel production. Over 350 oil-bearing crops are available as potential biofuel feedstock but soybean (*Glycine max*), rape seed (*Brassica napus*), sunflower (*Helianthus annuus*), and oil palm fruit (*Elaeis guineensis*) are among the few which had been commercialized (Demirbas, 2005).

Extraction of oil from oilseeds is predominantly by solvent and mechanical extraction. Solvent

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extraction of edible oil from their seeds, i.e., peanut (*Arachis hypogaea*), soybean, corn (*Zea mays*), sunflower, and palm kernel using hexane as solvent has been commercialized (Naksuk *et al.*, 2009). However, because of the volatility of hexane the process requires expensive equipment to ensure workers safety. United State Environment Protection Agency (2005) classified hexane as a hazardous air pollutant and vegetable oil extraction plants being a major contributor. Moreso, per tons of oil extracted, it is estimated that 0.7 kg of hexane is usually released into the environment.

Exposure to this hazardous compound at 125 ppm for three months causes peripheral nerve damage, muscle wasting, and atrophies (Pauzan *et al.*, 2013). Owing to this, alternative technologies are needed to eliminate the use of toxic solvents including hexane (Tyson *et al.*, 2004). Oil extraction by solution of aqueous surfactant is one of the promising alternatives. Few researchers have demonstrated the feasibility of application of aqueous-based oil extraction (Hagenmaier, 1974; South well and Harris, 1992; Evon *et al.*, 2007). Wu *et al.* (2011) used enzyme-assisted aqueous extraction to demulsified oil-rich emulsion and obtained 88% yield out of the 90% total oil available. Raman *et al.* (2003) demonstrated the feasibility of formation of micro emulsion by mixture of non-ionic surfactant with palm kernel oil and its derivatives. The principal salts used are NaCl, CaCl₂, and KCl. Monovalent salts are, however, preferred since divalent ions can cause surfactant precipitation (Negin *et al.*, 2017). Salts impact the electrostatic repulsions of the head groups in the case of ionic surfactants (Doan *et al.*, 2003) but also the steric repulsion between tails (He *et al.*, 2014).

In all those studies, extractions that are micro-emulsion based were evaluated as an alternative to hexane, for the extraction of vegetable oil and the approach is considered a clean technology since surfactants utilized for the extraction are biodegradable and non-toxic. This method is

based on the formation of micro emulsion induced by reduction of surface tension between the oil vegetable seeds and the aqueous extracting system. The reduction in the surface tension occurs as a result of inclusion of the surfactant in the aqueous medium.

Edible oil that contains triglycerides may be difficult to form a micro emulsion with, but this problem can be minimized by selecting a suitable surfactant extended and lipophilic linker (Komesvarakul *et al.*, 2006; Do and Sabatini, 2010). Hsu and Nacu (2003) reported the behavior of soybean oil in a water emulsion stabilized by non-ionic surfactant. Micro emulsion formation of eucalyptus oil by mixed non-ionic surfactant, polyoxyethylene (23) Lauryl ether (Brij 35) and anionic surfactant (AOT) using butanol alcohol as linker was reported (Rajib and Bidyut, 2005). Mixed surfactant systems are more efficient than solo surfactant systems (Salager *et al.*, 2019).

In order to prevent competition in using oil for food or fuel, a feedstock with less competition on food such as palm kernel seed is more attractive. In addition, oil extraction from palm kernel using mixed aqueous solution of sodium dodecyl sulphate (SDS) and polyoxyethylene (23) lauryl ether (Brij 35) has not been reported in the literature to the best of our knowledge.

Materials and Methods

The Biomass was sourced from a local oil palm plantation in Minna, Niger State, Nigeria. Surfactants used are: Sodium dodecyl sulphate, SDS (anionic surfactant), 98% purity; polyoxyethylene (23) Lauryl ether (non-ionic surfactant), 95% purity; and sodium chloride (an electrolyte). All were obtained from Sigma Aldrich, UK and used as received.

Sample Preparation

De-hulled Biomass was washed to remove all dirt and unwanted substances. After which it was oven-dried at 100°C, thereafter, it was grinded into granules of particle size ≤ 0.5 mm. The Biomass

was kept inside a desiccator to avoid adsorption of moisture.

FT-IR characterization of the oil extracted

The functional groups present in the extracted oil using hexane, petroleum ether and aqueous solution of mixed sodium dodecyl sulphate (SDS) and BRIJ-35 were identified by FT-IR spectroscope in the range of 4,000–500 cm^{-1} using FTIR-8400S, Shimadzu. The oil samples were mixed with oven-dried spectroscopic grade KBr and pressed into a disk (Bakar *et al.*, 2016).

Oil Extraction

Solvent based Extraction

50g of the grinded Biomass seed was weighed using a digital weighing balance and then wrapped with a filter paper. Then it was inserted into the extraction chamber (thimble) of the Soxhlet extractor. The solvent (hexane or petroleum ether) was filled into the 500ml round bottom flask and the extraction was conducted at 50°C for 24 h (Pauzan *et al.*, 2013). After the extraction, the solvent was evaporated from the solvent-oil mixture until constant mass. All extraction experiments were conducted in duplicates. The oil yield was computed using equation 1 (Pauzan *et al.*, 2013). The above procedure was repeated using petroleum ether as extraction solvent.

$$\text{Extraction yield} = \frac{\text{Mass of Extracted oil}}{\text{Mass of Biomass}} \times 100\% \quad \text{Eq. 1}$$

Mixed surfactant-based Extraction

1g of the grinded Biomass seed with particle size $\leq 0.5\text{mm}$ was weighed using a digital weighing balance. After which, it was mixed with 20 mL of aqueous surfactant solution concentrations varied from 0.2163-0.2595 wt % SDS; concentration of BRIJ 35 was fixed at 0.5995 wt %. The concentration of Sodium chloride was varied from 0-1wt% while liquid/solid ratio was varied from 4-10 ml/g to investigate their respective effects on oil extracted yield. The mixture was allowed to agitate on a rotary shaker at 150 rpm for a

specified contact time (0-35min) (Pauzan *et al.*, 2013). At the end of each contact time, the mixture was centrifuged to separate the surfactant and the extracted oil from the residual solid. The efficiency of the oil extracted was calculated using equation 1.

Results

Effect of SDS concentration on oil yield

Fig 1 shows the effect of SDS concentration on oil yield. There was an increment in oil yield from 30 to 38% as the concentration of SDS increased from 0.2163 to 0.2379 w%. The oil yield reduced to 30.5% as a result of further increase in SDS concentration from 0.2379 to 0.2595 w%. This result suggests that there was an increment in oil extraction yield as the SDS concentration increased owing to reduction in interfacial tension caused by increase in micelle concentration. The oil yield peaked at 0.2379 wt % SDS concentration and further increase in concentration reduced oil yield possibly due to solubilization of the free oil into the micelle (Pauzan *et al.*, 2013).

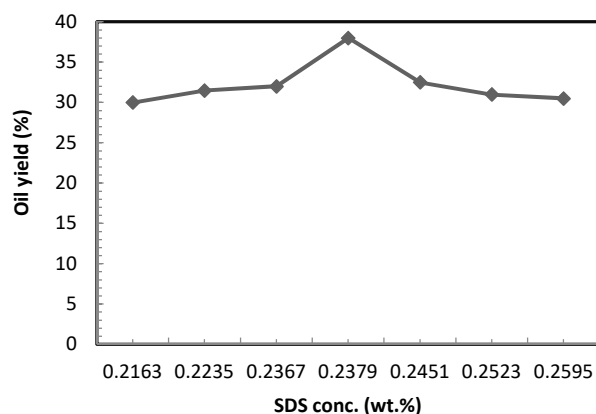


Fig 1: Graph of efficiency against Anionic surfactant concentration

Conc. of surfactant (0.2163-0.2595) wt. %, contact time (1min), grain size (0.212mm), mass of seed (1g), temperature (25 °C), agitation (150rpm).

Effect of mixed aqueous SDS and BRIJ 35 on oil yield

Fig. 2 shows the effect of mixed SDS and BRIJ-35 on oil yield. BRIJ-35 (non-ionic surfactant) concentration was fixed at 0.5995 wt.% while the concentration of SDS was varied from 0.2163 to 0.2595wt.%. BRIJ-35 addition to SDS increased the oil yield moderately from 53 to 56 % compared to a maximum of 38% oil yield obtained when 0.2379 wt.% SDS was used alone. The interaction between the anionic surfactant SDS at 0.2379 wt.% and 0.5995wt.% BRIJ-35 caused an increase in the yield as shown in Fig. 2. This trend could be as a result of inclusion of BRIJ-35 to SDS reduced the interfacial tension more than SDS alone and thereby caused an increase in liberation of the oil from the seed. Again, further increase in the concentration of SDS above 0.2379 wt% resulted into decrease in oil yield due to solubilization of the extracted oil into the micelles formed by mixture BRIJ-35 and SDS (Pauzan *et al.*, 2013).

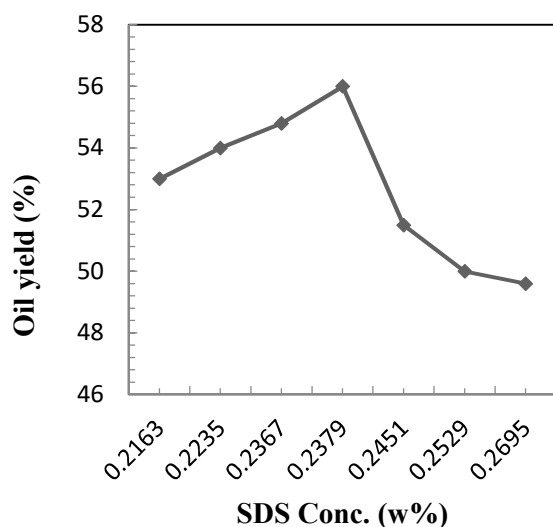


Fig. 2: Effect of SDS concentration on oil yield

SDS conc. (0.2163-0.2695 w%), BRIJ-35 conc. (0.5995w%), contact time (1min), grain size (0.212mm), mass of seed (1g), temperature (25 °C), agitation (150rpm).

Effect of sodium chloride on oil yield

Fig. 3 shows the effect of NaCl concentration on oil yield. The concentration of the salt was varied from 0 to 1 w% while SDS concentration was fixed at 0.2379 w% and BRIJ-35 at 0.5995w%. There was a moderate increase in oil yield to 59% at 0.3wt% concentration of salt, as a result of the addition of sodium chloride to the mixed surfactant solution. Sodium chloride is an electrolyte and helps in lowering the interfacial tension of surfactant solution (Naksuk *et al.*, 2009). It usually decreases the Hydrophile-Lipophile Balance (HLB) of an ionic surfactant system. Decrease in HLB of the system decreases interfacial tension suggesting why the oil yield slightly increased. However, excessive increase in the concentration of NaCl above 0.3 wt.% resulted in a decrease in the oil yield possibly owing to solubilization of the oil extracted into the surfactant micelle as shown in Fig. 3

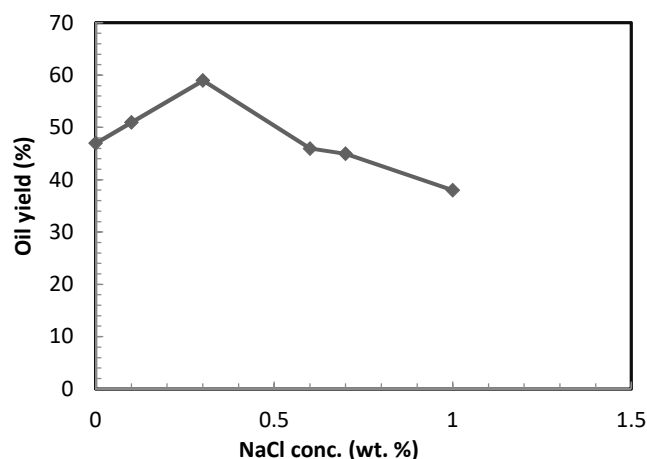


Fig. 3: Effect of NaCl on oil yield

Conc. of SDS surfactant (0.2379 wt.%), Conc. of BRIJ-35(0.5995 wt.%), Conc. of NaCl (0-1.0 wt. %), Contact time (1min), grain size (0.212mm), mass of seed (1g), temperature (25°C), agitation (150rpm).

Effect of contact time on oil yield.

Fig. 4 illustrates the effect of contact time on the oil yield. Increase in the contact time produced positive effect on the oil yield. Oil yield increased from 63 to 70.5% as the contact time increased from 1 to 25 min. However, further increase in contact time to 35 min steadily decreased the oil yield to 67.6%. This result conforms to the findings of Naksuk *et al.* (2009) where they obtained maximum oil yield in 30 min with further increase in contact time caused decrease in oil yield. The reduction in the oil yield at high contact time could be as a result of solubilization of the free oil extracted into the micelle.

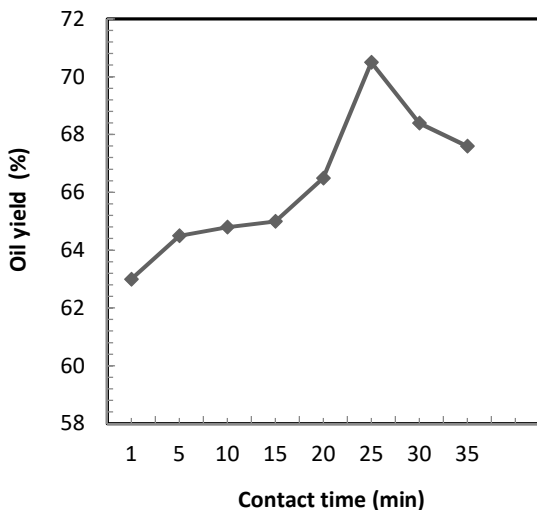


Fig. 4: Effect of contact time on oil yield.

Conc. of SDS surfactant (0.2379 wt.%), conc. of BRIJ-35(0.5995 wt.%), Conc. of NaCl (0.3 wt.%), contact time (1-35min), grain size (0.212mm), mass of seed(1g), temperature (25°C), agitation (150rpm).

Effect of liquid/solid ratio on oil yield

Fig 5 illustrates the effect of liquid-solid ratio on the oil yield. There was an oil increase in oil yield from 65 to 78.4% as the liquid-solid ratio increased from 4 to 10 ml/g. Further increase in the liquid/solid ratio to 12 ml/g caused slight reduction in the oil yield to 76 %. This result

conforms to other findings (Witthayapanyanon *et al.*, 2006). Again, the decrease in the amount of oil extracted at the highest liquid/solid ratio could be as a result of lower ineffective mixing, since the agitation rate was fixed at 150 rpm. In addition, excessive high liquid/solid ratio could reduce particle collision, resulting to low oil yield.

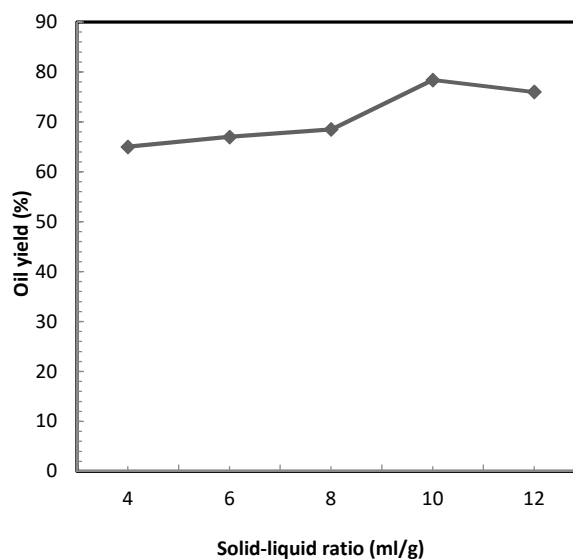


Fig. 5: Effect of solid-liquid ratio on oil yield

Conc. of SDS surfactant (0.2379 wt.%), Conc. of BRIJ-35(0.5995 wt.%), Conc. of NaCl (0.3 wt.%), Contact time (25min), grain size (0.212mm), mass of seed (1g), temperature (25°C), agitation (150rpm).

Mixed aqueous surfactant versus solvent based extraction

Solvent-based extraction method using petroleum ether and hexane was done once optimum conditions were obtained by extraction using mixed aqueous surfactant solution. A 50.0 g of the Biomass seed was used and the extraction was run for a period of 24hrs to get all the extractable oil from the seed. Petroleum ether and

hexane were used in this extraction to ascertain the solvent that will produce the highest yield since both are favorable in extracting oil. The average extraction efficiency using hexane and petroleum ether are 82% and 81% respectively. At optimum extraction conditions for the aqueous surfactant extraction, 0.2379 w% SDS, 0.5995 w% BRIJ-35, 0.3 w% NaCl, 10 ml/g liquid/solid ratio at 25 min were utilized to obtain 78.4% yield. Although the oil yield obtained using solvent based system is slightly higher than surfactant-based extraction, it is interesting to note that the surfactant-based extraction required 25 min to achieve the maximum 78.4% yield while hexane or petroleum ether used 24 h. to extract the maximum 81% and 82 respectively.

FT-IR characterization of the oil extracted

Fig. 6 illustrates the superimposed FT-IR spectra of the extracted oil using hexane, petroleum ether and aqueous solution of mixed sodium dodecyl sulphate (SDS) and BRIJ-35. The graph shows that there is no significant difference from the superimposed spectra suggesting that the mixed surfactant did not affect the quality of the extracted oil.

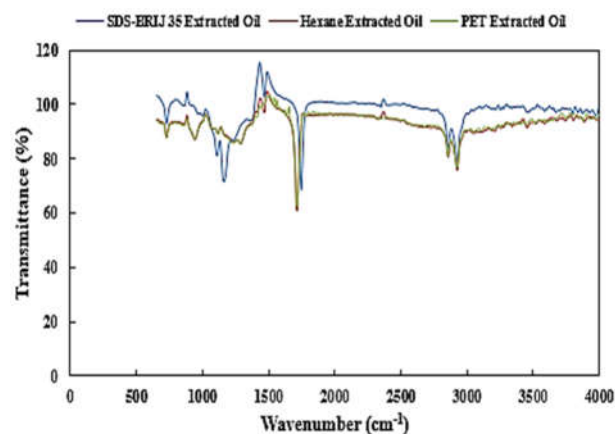


Fig. 6: Spectrum of and SDS vs BRIJ-35 Hexane, Petroleum ether superimposed.

Table 1: Major peaks of the FT-IR Spectrum of the oi extract

Wave number (cm-1)	Assignment	Reference
2924	Methylene C-H stretch	Coates (2000)
2916	Methylene C-H stretch	Coates (2000)
2862	Methylene C-H stretch	Coates (2000)
1736	C=O carbonyl functional group	Coates (2000)
1713	C=O carbonyl functional group	Coates (2000)
1465	Methylene C-H bending	Coates (2000)
1150	C-O stretch	Coates (2000)

Table 1 shows the major peaks and the assignments for the spectra. The peaks at 2924, 2916 and 2862 cm^{-1} is assigned to methylene C-H stretches while the peak at 1465 cm^{-1} is assigned to methylene C-H bending. The peaks at 1736-1713 cm^{-1} are assigned to carbonyl functional groups. The peak at 1150 cm^{-1} is assigned to C-O stretch originated from C-O-C of the carboxylic acid bonded to glycerol backbone of the triglyceride. All the functional groups are those expected from triglyceride which is the major components of the extracted oil.

Conclusions

This research demonstrates the possibility of using a mixed aqueous surfactant solution of SDS and BRIJ-35 as alternative to organic solvent based extraction for extracting oil from palm kernel. At optimum conditions for the aqueous mixed surfactant extraction (0.2379 wt.% SDS, 0.5995 wt.% BRIJ-35, 0.3wt.% NaCl, 10 ml/g liquid/solid ratio, 25 min contact time at 25°C), a maximum 78.4% oil yield was obtained. In contrast, at 50°C for 24hr, a maximum of 81% and 82% oil yield were obtained respectively when hexane and petroleum ether were used as oil extracting solvents. Addition of BRIJ-35 and NaCl to the SDS aqueous solution improved the oil yield.

Although the extraction efficiency is lower than extraction using solvent, the process requires shorter time and lower temperature than solvent-based extraction method. The extraction technology studied is more environmentally friendly. However, the challenge in aqueous mixed surfactant oil extraction is solubilization of the extracted oil inside the surfactant making separation difficult at high surfactant concentration. Therefore, low mixed surfactant concentration is suggested to reduce separation difficulty and further screening of more surfactants for this process is recommended.

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Competing Interest

I hereby declare that the author has no affiliations or financial involvement with anybody or organization with a financial interest or conflict with the subject or material discussed in this manuscript

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