Chemical Science International Journal

Comparative Study of Cashew Nut Shell Liquid and a Commercial Demulsifier for Treating Crude Oil Emulsions

C. O. Victor-Oji1*, U. J. Chukwu2 and O. Akaranta1

1 World Bank Africa Center of Excellence for Oilfield Chemicals Research, University of Port Harcourt, Nigeria. ² Department of Pure and Industrial Chemistry, University of Port Harcourt, Nigeria.

Authors' contributions

This work was carried out in collaboration among all authors. Author COVO designed the study, performed the data analysis, wrote the protocol and wrote the first draft of the manuscript. Author UJC managed the analyses of the study. Author OA managed the literature searches. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/CSJI/2019/v28i430148 *Editor(s):* (1) Dr. Yunjin Yao, Professor, Department of Chemical Engineering, School of Chemistry and Chemical Engineering, Hefei University of Technology, Tunxi Road 193, Hefei 230009, China. *Reviewers:* (1) Oloro Obarhire John, Delta State University, Abraka, Nigeria. (2) G. Sujaykumar, Visvesvaraiha Technological University, India. Complete Peer review History: http://www.sdiarticle4.com/review-history/53544

Original Research Article

Received 20 October 2019 Accepted 26 December 2019 Published 02 January 2020

ABSTRACT

Cashew Nut Shell Liquid (CNSL), a natural phenolic liquid extracted from locally sourced raw materials, and Phasetreat-4633 (a commercial demulsifier) were diluted with xylene and butanol and used as demulsifiers in the treatment of crude oil emulsions. Laboratory simulated crude oil emulsions dosed with different concentrations of CNSL and PT-4633 were the basis for comparison of demulsifier performance via the bottle test method.

Different factors affecting demulsification efficiency such as; water content, demulsifier concentration, and solvent type were investigated at 10%, 30%, and 50% water content. The data obtained showed that the rate of water separation increases with increasing concentration and water content of the demulsifiers and emulsion respectively. Among the demulsifier-solvent combination employed in this study, Phasetreat-4633 in butanol recorded the most efficient water separation with optimal (100%) separation recorded after 5 minutes at 40 ppm and 50 ppm concentration, 50% water content and 60℃.

**Corresponding author: E-mail: victor.cynthia@aceuniport.org, victor.cynthia@aceuniport.edu.ng, cynthia.anyanwu@uniport.edu.ng;*

From the obtained results, the investigated demulsifiers have great potential with butanol as a solvent in the resolution of crude oil emulsions in shorter times. This behavior can be attributed to the synergetic effect of butanol as a solvent. Hence, butanol can be regarded as a better solvent substitute to xylene, due to its attributive synergetic effect, low cost and toxicity levels, unlike xylene which is more toxic and expensive.

Keywords: Bottle test; Cashew Nut Shell Liquid (CNSL); crude oil emulsions; Demulsification efficiency demulsifiers.

1. INTRODUCTION

Crude oil is rarely formed unaccompanied from reservoirs as a compound mix of hydrocarbons and formation water resulting in the formation of an emulsion, in which water is dispersed as fine droplets in the oil [1,2]. The emulsions formed are undesirable and its resolution is one of the major challenges encountered by production chemists and engineers in the course of crude oil production [3]. Oilfield emulsions can occur either as water-in-oil (W/O), oil-in-water (O/W) or water and oil in water (W/O/W) multiple emulsion systems. However, most occurring oilfield emulsions are of the W/O type [1]. Several crude oil emulsion treatment methods exist which comprise; chemical treatment, thermal, mechanical, electrical methods [4]. However, the most common method of emulsion treatment is dosing with chemicals.

Demulsifier chemicals, which are referred to as 'Chemical additives' are special surface acting agents which once added to the crude oil emulsion, moves to the water-oil interface [5]. They act by destabilizing the interfacial film [3]. To ensure the optimal performance, a demulsifier should be able: dissolve in the oil phase, partition into the water phase; and have sufficient concentration to break the interfacial film of the crude oil emulsion, etc. Commercial demulsifier formulations that exist include: Phasetreat-4633 from Clariant oil services, VX7079 from ESSO, etc. [6,7].

Cashew is grown abundantly in Nigeria mainly for its kernel whereas the shell is generally regarded as waste [8]. Cashew Nut Shell Liquid (CNSL) is a by-product of the cashew-nut industry and is extracted from the spongy mesocarp of cashew nut (*Anacardium occidentale L.*) shell by a number of techniques, usually involving pyrolysis or solvent extraction of the shells [9]. It is a rich source of phenolic lipids, has anacardic acid, cardol, cardanol and 2 methyl cardol as its components (Fig. 1).

[10,11,12,3,13]. The availability of CNSL globally is approximately 50 kilotons per annum and as an agricultural by-product of the cashew nut production, it is one of the major economic sources of naturally occurring phenols and is regarded as a versatile and valuable raw material for polymer production [14]. CNSL is a low-cost and renewable material that can be used to replace phenol with a good advantage. It appears as a dark-brown viscous oil, with a characteristic smell, quite unlike other vegetable oils [15].

Fig. 1. Chemical composition of CNSL: (a) anacardic acid, (b) cardanol, (c) cardol, (d) 2-methylcardol [11,12]

There are two types of CNSL depending on the extraction method; Solvent extracted/Natural CNSL and Technical CNSL. Cold-extracted CNSL using low-boiling solvents is termed natural CNSL. It is composed of; anacardic acid (60-65%), cardol (15-31%), cardanol (10-21%) and trace amounts of methyl-cardol while Technical CNSL consist of (60- 65%) cardanol, (15-20%) cardol (1-2%), anacardic acid, (0.3- 10%) polymeric material [11,16]. Although CNSL has found a number of industrial applications, it is still highly under-utilized in the petroleum industry, especially in Nigeria where it has very low economic value [13]. The constituents of CNSL are structurally suited for use as surfaceactive agents for petroleum industry-related applications, and it also possesses certain outstanding properties which makes it suitable for use in countless applications [10,12]. The advantages of using CNSL as a demulsifier among others include; its low cost, availability, sustainability, and its bioavailability in nature, thus making it a replenishable raw material.

The laboratory bottle testing procedure is not a standard method, but based on trial and error and is usually done under static and controlled conditions, remains the only dependable method for the evaluation, selection, and screening of prospective demulsifiers for a particular well or field [6]. This procedure gives an estimate of treating temperatures and water retention times that can be used for design and oilfield operations [17,18]. The suitable temperatures considered for a laboratory-scale demulsification experiment lie between 50℃ to 70℃, which is similar to the actual operational temperature [19,20].

Some studies have investigated the influence of solvents, mainly aromatics, such as; benzene, toluene, xylene, ethylbenzene, tert-butyl benzene and cymene, on emulsion stability and results have shown that the most aromatic solvents (with higher aromatic carbon content) are more effective in demulsification of emulsions [21,22]. The disadvantage of having organic solvents in a demulsifier formulation include increased cost, flammability, and toxicity among others, therefore, a demulsifier formulation which does not include organic solvents would represent an advance in the art of demulsification [23].

This paper is focused on the comparative and investigative study of the effectiveness of a commercial demulsifier and CNSL using two selected solvents, xylene and butanol, in the treatment of crude oil emulsions.

2. MATERIALS AND METHODS

2.1 Materials

2.1.1 Crude oil

Medium heavy crude oil was sampled from one of Shell Petroleum Development Company (SPDC) flow stations located within the Niger Delta region, labeled accordingly and dispatched to the laboratory for further analysis. Its

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physicochemical characterization is as shown in Table 1.

2.1.2 Seawater

Seawater was sampled from the Gulf of Guinea via the SPDC Bonga platform in the Niger Delta region, labelled accordingly and dispatched to the laboratory for further analysis. Its physicochemical characterization is as shown in Table 2.

2.1.3 Cashew Nut Shell Liquid (CNSL)

Whole cashew nuts were procured from Ihube village in Okigwe Local Government Area of Imo State, Nigeria. The nut was cut lengthwise, dekernelled and crushed using a hydraulic press (to increase the surface area for extraction). 300 g of crushed cashew nut shell was loaded into a Soxhlet extractor and extracted by refluxing with acetone until the solvent becomes clear in the extraction thimble. The extracted natural cashew nut shell liquid was recovered from the solvent by distillation technique.

2.1.4 Chemicals

The chemicals used include; Industrial grade acetone, xylene and butanol (Analar grade Aldrich Chemicals). All chemicals were used as received without further purification.

2.1.5 Demulsifiers

The demulsifiers used in this study include; Cashew Nut Shell Liquid (CNSL) and Phasetreat (PT) 4633, the commercial demulsifier used in most SPDC flow stations.

2.2 Methods

2.2.1 Cashew Nut Shell Liquid (CNSL) characterization

The CNSL has been characterized and pH, specific gravity (ASTM D-1298-12b), acid value, iodine value (wijs method), kinematic viscosity (40ºC & 100ºC) (ASTM D455-12) and dynamic viscosity over a temperature range of 10 – 60ºC using Ofite® 900 viscometer have been determined [24]. FTIR analysis was conducted with an Agilent spectrophotometer scanning in the 4000 – 700 $\rm cm^{-1}$ range.

2.2.2 Crude oil characterization

Specific gravity and API gravity: Specific (60/60ºF) of the crude oil sample was determined according to ASTM D1298-12b using a hydrometer and API gravity calculated as follows:

$$
API\ gravity = \frac{141.5}{Specific\ Gravity} - 131.5
$$

Kinematic viscosity: Kinematic viscosity was determined at 40ºC and 100ºC according to ASTM D455-12, using a Stanhope-Seta KV-8 Viscometer bath.

Pour point: Pour point of the crude oil was determined using a Stanhope-Seta Pour Point refrigerator according to ASTM D5853-17a. The crude oil was heated to 45℃ and the test jar containing the oil, placed in the cooling jacket of the pour point refrigerator, the test jar was gently extracted from the cooling jacket to check for flow at every 3ºC decrease in temperature repeatedly, until the pour point is reached.

Water Cut: Water-cut was determined by Dean-Stark distillation according to ASTM D4006-11. Homogenize sample by agitation and measure 100 ml of the sample into a round bottom flask. Add an equivalent amount of xylene to the flask, fix the dean and stark receiver trap to the condenser and heat for one hour and record the reading of water collected in the trap. The water cut is then estimated as follows:

Water cut (
$$
\%
$$
) = $\frac{Volume\ of\ water\ collected\ in\ the\ trap}{Volume\ of\ sample}$
× 100

Sulphur content: Sulphur content of the crude was determined according to ASTM D4292-16 using a Horiba Sulphur-in-oil analyzer.

Base, Sediment and Water (BS&W): The BS&W content of the crude was determined according to ASTM D4007-11 by Centrifuge method. 50 ml of the sample is introduced into a centrifuge tube followed by the addition of an equivalent amount of xylene. 5 drops of demulsifier is added and agitated 10 times in a to and fro motion. The tube is placed in a thermostatic water bath set to 60ºC for 15 minutes after which it is centrifuged for 10 mins and the readings taken.

2.2.3 Seawater characterization

Resistivity and conductivity: The electrical conductivity of the seawater was determined according to ASTM D1125-14 using a YSI 3200 conductivity instrument and the electrical resistivity calculated as follows:

$$
Resistivity = \frac{1}{Conductivity}
$$

Total Dissolved Solids (TDS): The total dissolved solids in the seawater was determined according to ASTM D5907-18. The crucible was preheated to constant weight at 180℃ and placed in the desiccator for two hours before weighing. 100 ml of the seawater is filtered through a 45μ filter paper into a receiving flask. The constant weight crucible is filled with the seawater filtrate and placed on a water bath. The filtrate is left to evaporate to dryness, oven dried and weighed. The total dissolved solids can be calculated thus:

Total Dissolved Solids, TDS (ppm) $\frac{1}{2}$ (weight of crucible + filtrate) – (weight of crucible) volume of sample \times 10⁶

Specific gravity and density: The specific gravity of the seawater was determined according to ASTM D1429-13 using a hydrometer and density calculated.

Ph: The pH of the seawater was determined according to ASTM D3875-03 using Thermo Scientific Orion Star A211 pH meter.

Salinity: The salt content of the seawater was determined according to ASTM D4458-15. Pipette an aliquot of the sample (0.1 ml) into a 25ml measuring cylinder, dilute to mark with distilled water and pour into a 50 ml Erlenmeyer flask. Add 0.25 ml of 5%w/v potassium chromate indicator and titrate with 0.0140N silver nitrate $(AgNO₃)$ solution to the end point (pinkish yellow or brick red). Record the volume of $AgNO₃$ required to the end point and calculate the chloride content and salinity as follows:

Chloride content, $Cl^-(^{mg}/_L)$ $=\frac{(mL AgNO_3)_{1} (mL)_{2} (mL)}{4}$ × molarity of $AgNO_3 \times 35450$ volume of sample

Salinity $\binom{mg}{L}$ = Cl⁻ \times 1.8066

2.2.4 Preparation of water-in-crude oil emulsions

Laboratory simulated water-in-crude oil emulsions were prepared by stirring the crude oils at high speed using a Hamilton Beach commercial mixer for 30 minutes with gradual

Parameter	Method	Value
Specific Gravity (60/60°F)	ASTM D 1298	0.8980
API Gravity@ 60°F	ASTM D 1298	26.0723
Kinematic Viscosity at 40℃ (c.St)	ASTM D455	13.6426
Kinematic Viscosity at 100℃ (c.St)	ASTM D 455	3.1469
Water cut $(\%)$	ASTM D 4006	0.0250
Sulphur content (wt.%)	ASTM D 4292	0.3348
Base, Sediment and Water (%)	ASTM D 4007	0.025
Pour Point (°C)	ASTM D 5853	-50

Table 1. Physico-chemical properties of crude oil samples

addition of seawater until both phases become completely homogenized. The emulsions were prepared at different mixing ratios of crude oil to water; 90:10, 70:30 and 50:50, respectively.

2.2.5 Demulsifier preparation

Five weight percent (5%w/w) of the demulsifiers were dissolved in 100 g volume equivalent of Xylene and Butanol respectively to give 50000 ppm stock solution. A 1000 ppm standard solution of the demulsifier was prepared by adding 2 ml of the stock solution in a 100ml volumetric flask and fill to mark with the respective solvents.

2.2.6 Bottle testing

The bottle test method was used to evaluate the efficiency of the demulsifier-in-solvent demulsifier-in-solvent combination in the simulated crude emulsions. The simulated crude emulsions were placed in a graduated Teflon-stoppered 100 ml bottle with demulsifier in xylene and butanol added at different concentrations of 10, 20, 30, 40 and 50ppm respectively. A blank was used for each set of experiment. The bottle was agitated 100 times in a 'to' and 'fro' motion (to mimic the natural mixing of crude oil and demulsifier in the flow station) and were immersed in a thermostatic water bath maintained at a temperature of 60℃, the average treating temperature in the field, with water seperation

observed for the initial 5 mins and subsequently at every 10 mins interval for a 3-hour duration. The demulsifier performance was assessed based on its water seperation rate as determined by percentage water seperation which is given as:

Water seperation $(\%)$

Volume of seperated water in ml $\frac{1}{\pi}$ $\frac{1}{\pi}$

3. RESULTS AND DISCUSSION

3.1 Crude Oil Characterization

The physico-chemical properties of the crude oil sample are presented in Table 1. The API
(American Petroleum Institute) gravity Petroleum Institute) gravity classification shows that the crude is a medium heavy crude oil.

3.2 Sea Water Characterization

The physico-chemical properties of the seawater sampled from the Gulf of Guinea via SPDC Bonga FPSO platform are presented in Table 2.

3.3 Characterization of CNSL

The physico-chemical properties of the extracted CNSL are presented in the Table 3.

The FTIR spectrum of CNSL shows		
characteristic peaks corresponding to the		
functional groups in anacardic acid (Fig. 2). A		
strong and broad absorption band is observed for		
the phenolic O-H group before the C-H		
stretching vibration of alkene which is at 3011.7		
cm^{-1} . There are bands at 2922.2 cm^{-1} and 2855.1		
$cm-1$ which is due to the C-H vibrations of		
methylene and methyl groups of the hydrocarbon		
chain respectively. The C=O of the acid attached		
to aromatic ring absorbs at 1699.7 cm^{-1} while		
1207.7 cm^{-1} is the C-O stretching vibration of the		
acid. The vibration at 1643.8 cm ⁻¹ is the C=C		
stretching of alkene, while the band at 1602.8		
cm^{-1} corresponds to aromatic C=C vibrations.		
Methyl C-H deformation vibrations occur at		
1449.9 cm^{-1} and 1300.8 cm^{-1} .		

Table 3. Physico-chemical properties of extracted CNSL [24]

3.4 Evaluation of Phasetreat (PT) 4633 and CNSL in Xylene and Butanol

Some factors which affect the demulsification efficiency were studied as follows:

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3.4.1 Effect of dosage/concentration

Several sets of experiments were carried to evaluate the effect of concentration on the demulsification process, as this parameter governs demulsifier adsorption at the interface. The results are presented in Figs. 3 to 12 for PT 4633 and CNSL in xylene and butanol at different water content and concentrations of 10-50 ppm at 60℃ respectively. The plotted data revealed that an increase in demulsifier concentration decreases the demulsification time which may be due to an increase in the rate of partitioning which further increases the adsorption of the demulsifier molecules at the emulsion interface
[25]. In Figs. 3-7, the demulsification [25]. In Figs. 3-7, the demulsification performance of PT-4633 demulsifier in xylene and butanol increases as the water content and concentration increases, with an optimal seperation of 100% observed at 40 ppm at a seperation time of 20mins and 40-50 ppm at a seperation time of 5 mins and 50% water content for PT-4633 in xylene and butanol respectively.

This behavior was also observed for CNSL in xylene and butanol however, CNSL in xylene exhibited an optimal performance of 60% at 20 ppm and 50% water content at a seperation time of 150 mins which was followed by a performance of 56% at 50ppm and 50% water content at a seperation time of 90 mins. Using CNSL in butanol, an optimal performance of 100% was observed at 50 ppm and 50% water content in 160mins which is presented in Figs. 8- 12. Generally, for all demulsifier types, as
demulsifier concentration increases. the concentration increases, the

Fig. 2. FTIR spectrum of the extracted Cashew Nut Shell Liquid

interfacial tension of the emulsion interface decreases, thus increasing the water seperation percentage [25].

3.4.2 Effect of solvent

To investigate the effect of solvents on the demulsification efficiency; the demulsifiers; PT-4633 and CNSL, were used in two solvents, xylene and butanol. The data obtained were compared and illustrated in Figs. 3-12 and it shows that butanol is a better solvent and attributes a positive synergetic effect, which may be by virtue of the hydroxyl -OH in butanol, in optimizing the demulsification performance.

Figs. 3(ii)-12(ii) show that data obtained with the demulsifiers in butanol was better than that of xylene (Figs. 3(i)-12(i) as it took less time to achieve optimal seperation under the same conditions. The time taken to complete water separation was 160 mins, 180 mins, 170 mins for PT-4633 in xylene and 150 mins, 50 mins, and 5min for PT-4633 in butanol at 50ppm and 10%, 30%, and 50% water content respectively as shown in Fig. 7(i) and 7(ii). On the other hand, the time taken to complete water separation with CNSL under the same conditions was 180mins, 160 mins, and 90 mins in xylene and 160mins, 180 mins, and 160 mins, in butanol with however more optimal seperation observed as seen in Fig. 12(i) and 12(ii). Solvent adsorption weakens and ruptures the interfacial film making coalescence rapid and leads to an increase in the rate of emulsion resolution. The minimum time taken for complete emulsion separation was obtained using butanol.

(ii)

Fig. 3. Water seperation (%) versus seperation time (mins) for Phasetreat-4633 demulsifiers in (i) xylene and (ii) butanol, respectively at 10 ppm, different water content and 60℃

(ii)

Fig. 4. Water seperation (%) versus seperation time (mins) for Phasetreat-4633 demulsifier in (i) xylene and (ii) butanol, respectively at 20 ppm, different water content and 60℃

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(ii)

Fig. 5. Water seperation (%) versus seperation time (mins) for Phasetreat-4633 demulsifier in (i) xylene and (ii) butanol, respectively at 30ppm, different water content and 60℃

(ii)

Fig. 6. Water seperation (%) versus seperation time (mins) for Phasetreat-4633 demulsifier in (i) xylene and (ii) butanol, respectively at 40ppm, different water content and 60℃

(ii)

Fig. 7. Water seperation (%) versus seperation time (mins) for Phasetreat-4633 demulsifier in (i) xylene and (ii) butanol, respectively at 50 ppm, different water content and 60℃

Fig. 8. Water seperation (%) versus seperation time (mins) for CNSL demulsifier in (i) xylene and (ii) butanol, respectively at 10ppm, different water content and 60℃

Fig. 9. Water seperation (%) versus seperation time (mins) for CNSL demulsifier in (i) xylene and (ii) butanol, respectively at 20ppm, different water content and 60℃

(ii)

Fig. 10. Water seperation (%) versus seperation time (mins) for CNSL demulsifier in (i) xylene and (ii) butanol, respectively at 30ppm, different water content and 60℃

Fig. 11. Water seperation (%) versus seperation time (mins) for CNSL demulsifier in (i) xylene and (ii) butanol, respectively at 40 ppm, different water content and 60℃

Fig. 12. Water seperation (%) versus seperation time (mins) for CNSL demulsifier in (i) xylene and (ii) butanol, respectively at 50ppm, different water content and 60℃

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3.4.3 Effect of water content

Water content in an emulsion plays an imperative role on demulsifier performance. The effect of increasing water content on demulsifier performance was studied at different concentrations and 10%, 30% and 50% water content respectively as illustrated in Figs. 3-12. The data obtained showed that the time taken for water seperation decreases with increasing water content and concentration of demulsifiers. This likely infers that as the demulsifier molecules migrate and their distribution on the emulsion interface occurs, there is a reduction of the interfacial layer potential which helps to break up the interface, releasing water droplets to coalesce leading to rapid water separation [26].

4. CONCLUSION

From the study, the following conclusion(s) can be deduced:

- The chemical structure of the demulsifiers and solvent, which contain hydroxyl groups may have played a role in increasing the partitioning which in turn enhanced demulsification performance.
- Increasing demulsifier concentration (from 10 ppm to 50 ppm) decreases the time taken to achieve optimal water separation.
- Increasing water content in a crude oil emulsion leads to increase in the rate of water seperation, which can be attributed to an increased thinning of the interfacial film.
- Optimal demulsification efficiency was obtained by PT-4633 and CNSL in butanol (100% water separation was obtained after 5 and 160 minutes respectively at 50ppm and 50% water content), which may be due to its synergetic effect.
- Though, the demulsification performance of CNSL is low compared to PT-4633, and considering the increasing production of cashew nut across the world, we can consider CNSL as a more sustainable, less expensive, and renewable feedstock for industrial use without its depletion.
- The results of the demulsification performance were compared with the existing demulsifier formulation (PT-4633 in xylene), which is in use in the fields. The data showed that the efficiency of the commercial demulsifier, PT-4633 in butanol, is better than the currently used demulsifier-solvent combination.

 Among the demulsifier-solvent combination employed in this study, Phasetreat-4633 in butanol recorded the most efficient water separation regardless of the cost. Thus, butanol can be regarded as a better solvent substitute to xylene. This is due to its attributive synergetic effect, low cost and toxicity levels unlike xylene which is toxic and expensive.

Though, the demulsification performance of CNSL is low compared to PT-4633, however, considering the increasing production of cashew nut across the world, CNSL is a more sustainable, less expensive, and renewable feedstock for industrial use as demulsifiers without its depletion and will suggest that further works be carried out on the economics and toxicity of the formulations with CNSL in comparison with that of the available commercial demulsifier(s) in the field.

ACKNOWLEDGEMENTS

The authors wish to acknowledge the Production Chemistry team, Shell Petroleum Development Company (SPDC), Nigeria for their support which led to the successful completion of this study.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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APPENDIX

Table 4. Demulsification efficiency for PT-4633 in xylene and butanol at 60℃

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