International Journal of Advances in Engineering and Management (IJAEM)
Volume 7, Issue 07 July 2025, pp: 783-792 www.ijaem.net ISSN: 2395-5252

Development of Local Demulsifier for the Treatment of Crude Oil Emulsion

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Date of Submission: 15-07-2025 Date of Acceptance: 25-07-2025

ABSTRACT

The presence of emulsion in crude oil presents a flow assurance problems, part of which is raising the bulk fluid's viscosity. Almost every part of the oil production and processing chain is susceptible to emulsions because of the ubiquitous presence of water in the production process. Water in crude oil may have unintended consequences such as increased corrosion rate, higher conductivity, and subtle leaching of additives. To reduce the corrosion rate and prevent catalyst poisoning at downstream processing facilities and to meet crude standards from storage to export, the emulsion needs to be treated to remove the distributed water and associated inorganic salts. The Niger Delta being one of the leading oil producing regions in Nigeria and in the world has remained dependent on foreign demulsifiers for the treatment of crude oil emulsions. Some of these chemicals may not be environmentally friendly due to their constituent elements which may not be readily biodegradable. Thus, there is the need for a readily biodegradable materials for the formulation of environmentally friendly demulsifier for the treatment of Niger Delta crude oil.From studies, most agro-based oils are rich in fatty acids and non-saturated fats that can be blended with other materials to produce a surface-active compound that can break the interfacial surface of the water droplets and coalesce with each other. Three demulsifier blends $X_{1,}$ X_2 and X_3 were prepared using different ratios of palm kernel oil, cashew nut oil, liquid soap, locally prepared starch and Calcium Hydroxide. Blend X_1contains 40% of palm kernel oil, 40% of cashew nut oil, blend X_2 contains 50% of palm kernel oil and 30% of cashew nut oil while blend X_3 contains 30% of palm kernel oil and 50% cashew nut oil. The percentage of liquid soap, starch, calcium hydroxide and water is the same in each of the three blends. All three blends were used as demulsifier individually to separate water droplets in oil and they individually performed optimally at 0.6 ml within 30 minutes in this work. The imported demulsifier used for comparison in this study is polydimethylsiloxane (PDMS) which took nearly 120 minutes at 1.0 – 1.4 ml to yield the same results as the locally prepared demulsifiers. A maximum of 15.5 ml resulting to 15.5 % of water was released from the crude oil emulsion using the locally blended demulsifiers. The quality of oil and water separated after demulsification with the various local blends shows that all the blends produced good oil quality with sharp oil - water interface and complete separation within 30 to 120 minutes. The results were in line with the specification allowable for the BS&W contained in crude oil for export and the local refineries as stipulated by the Nigerian Upstream Petroleum Regulatory Commission (NUPRC)

Keywords: Emulsion, Demulsifier, Palm kernel oil, Cashew nuts oil, Biodegradable materials.

I. INTRODUCTION

The production of hydrocarbon from the reservoir through the wellbore to the surface production/process facilities is usually a multiphase flow with associated flow assurance problems [1]. Flow assurance in terms of production, covers all methods to ensure the safe and efficient delivery of hydrocarbons from the well to the receiving facilities [3], [5]. Flow assurance is the ability to produce reservoir fluids economically from the reservoir to the production facilities over the life of the field and in all conditions & environments [2]. Examples of flow assurance problems are wax, emulsion, sand, asphaltene etc. The focus of this study is on the treatment of crude oil emulsion. To reduce corrosion and catalyst poisoning at downstream processing facilities and to meet crude standards from transportation, storage and export stages, the emulsion must be treated to remove the

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distributed water and associated inorganic salts [12].

Methods of Treating Emulsions

Ajienka listed seven fundamental approaches to treating water-in-crude-oil emulsions, these includes: heating, gravity settling, electrical, dilution, centrifugation, chemical (the subject of this research) and filtration. The decision to use a thermal approach, also known as heat treatment, for emulsion breaking, is typically dependent on the treating facility's economics as a whole [2], [4] and [5].

Yogesh et al. (2022) listed a variety of demulsification methods, including mechanical, electrical, thermal, membrane, and chemical processes. The extraction of crude oil from emulsions requires a fast and low-cost method. Demulsification is mostly accomplished using chemical techniques today. To speed up the water-separating process, oil corporations are investigating oil from plants as a potential demulsifier ingredient. Recently, green demulsifiers made from organic materials have become popular [26], [27], [28], and [29].

As Saad et al. (2020) pointed out, the eco-friendliness of materials sourced from natural products has garnered a lot of attention, which means that the need to replace petroleum-based oil field chemicals with plant-based materials has also received significant attention and it is the purpose of this research to develop a method for synthesizing a surfactant that could be used to break up water-in-oil emulsions [21], [22], and [25].

Significance of The Study

Over the years, the over reliance of Nigeria on imported materials/chemicals in the oil and gas industry which are capital intensive calls for concern. Hence, the drive for local content application in the oil and gas industry by the Federal Government of Nigeria cannot be overemphasized. This is in line with this study to formulate a locally made demulsifier for the treatment of crude oil emulsion. Therefore, this study is very important to treat emulsion formation in crude oil production, transportation, and storage. Also, when the crude is not well treated, it affects the quality of the crude and downstream processing costs.

Scope of the Study

This present study is focused on oil in water emulsion from crude within Niger Delta fields. The proposed demulsifier will be developed completely with locally sourced materials that are cheap and environmentally friendly. The prepared demulsifier will be tested at ambient temperature to ascertain its efficacy and its general application in the oil and gas industry. To validate its performance, it will be compared with an imported demulsifier already in use in the industry.

II. MATERIALS AND METHODS

In order to formulate a local demulsifier that would be capable of breaking water in oil emulsion, an oil soluble demulsifier is required. Most agro-based oils are rich in fatty acids and non-saturated fats that can be blended with other materials to produce a surface-active compound that helps to break the interfacial surface of the water droplets and coalesce with each other. Polydimethylsiloxane (PDMS) is mostly used as component in silicone grease and other silicone-based lubricants, as well as in defoaming agents, mold release agents, damping fluids, heat transfer fluids, polishes, cosmetics, hair conditioners and other applications.A silicone oil-in-water emulsion concentrate (10% w/v) is prepared by homogenizing silicone oil in water (at a water temperature of about 2-5° C.) for 5 minutes. The silicone oil concentrate (10% w/v) is added to water at 2-5° C to provide a final silicone oil-in-water emulsion with a concentration of 0.8% w/v The materials used for the experiment are listed below:

i. Palm Kernel Oil (PKO)
ii. Cashew Seed Oil (CSO)
iii. Crude Oil
iv. Polydimethylsiloxane (PDMS)
v. Calcium Hydroxide ($Ca(OH)_2$)
vi. Liquid Soap
vii. Starch

Extraction of Palm Kernel Oil (PKO)

The palm kernel oil processing starts with shelling of the dried palm kernel. The shells are usually removed by hitting the hard nuts between two stones to crack the shells and then simultaneously separating the nuts from the shells. After the shelling process is completed, the nuts are sun dried, depending on the prevailing weather conditions, normally a 24-hour sun drying is enough for the nuts to dehydrate its moisture content. The nuts are then fried in a metallic container for about 30 to 45minutes in a moderate heat supply. The fried palm kernel nuts are then pounded in a mortar or grounded to paste with a motorized grinder. The grounded nuts are then

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mixed with water to obtain a paste like mixture which is then heated to release the palm kernel oil. The released oil is periodically skimmed from the surface of the heated paste into a container.

Extraction of Cashew Seed Oil (CSO)

Three viable methods for extracting oil from cashew nuts are mechanical, roasting, and solvent extraction. Solvent extraction yields 20 to 25 percent oil by volume and is considered the most practical method. The nuts were frozen overnight to harden the shell. Then, lightly pounding along the axis where the shell halves meet, the nuts were split in half. This process yielded intact nuts. In order to extract CSO from cashew nuts, the cashew nuts are lightly fried in a container and introduced into a hydraulic press which screw-press the nuts. The oil is collected in a container

Table 2.1 Percentage composition of fatty acids in Palm Kernel Oil and Cashew Seed Oil

Fatty acid	**Palm Kernel Oil(%)**	**Cashew Seed Oil(%)**
Caproic Acid (6:0)	0.2	-
Caprylic Acid (8:0)	3.3	-
Capric Acid (10:0)	3.5	-
Lauric Acid (12:0)	47.8	-
Myristic Acid (14:0)	16.3	-
Palmitic Acid (16:0)	8.5	9.85
Palmitoleic Acid (16:1)	9.6	0.26
Stearic Acid (18:0)	2.4	10.93
Oleic Acid (18:1)	15.4	60.87
Linoleic Acid (18:2)	2.4	17.33
Arachidic Acid (20:0)	0.1	0.76
Total Saturated Fatty Acid	82.1	21.60
Total Mono-unsaturated Fatty Acid	15.4	78.14
Total Poly-unsaturated Fatty Acid	2.4	-

Crude Oil Sample Properties

The specific gravity and API gravity of the crude oil were determined using the density bottle method. In order to determine the specific gravity of the crude oil sample, the density of the sample has to be determined. The density of the crude oil was obtained using experimental procedures outlined below and the value was recorded. The density of any fluid is the ratio of its mass per unit volume. The density of the crude oil sample was determined with a known volume of the sample and it is expressed as:

$$\text{Density}(\rho) = \frac{\text{Mass}}{\text{Volume}} \tag{1}$$

$$\text{Specific Gravity (g)} = \frac{W_1 - W_2}{W_3 - W_2} \tag{2}$$

$$\text{API Gravity} = \frac{141.5}{\gamma} - 131.5 \tag{3}$$

Density of Crude Oil Using Density Bottle Method

The density of a substance is expressed as the ratio of its mass to its unit volume. The formula for density is shown in equation (1) above. In the laboratory, it is expressed in units of gram per cubic centimeter or gram per milliliter (g/ml). The data correlated for light, medium and heavy crude oil samples from different locations in Niger Delta show densities ranging from 0.813-0.849 g/ml, 0.866- 0.886 g/ml, and 0.925- 0.956 g/ml.

API Gravity of Crude Oil Using Density Bottle Method

API gravity is short for American Petroleum Institute gravity, an inverse measure that is used to determine the weight of petroleum liquids in comparison to water. If a liquid has API gravity of more than 10 it is considered a light oil that floats on water. API gravity essentially measures the relative density of petroleum liquid and water. It is primarily used to evaluate and contrast the relative densities of petroleum liquids. In mathematical terms API gravity has no dimensions. However, the measure is gradated in degrees.

Pour Point of Crude Oil Sample Using Test Tube Method

The pour point is a fluid property which is often used in flow assurance applications. The pour point of crude oil is the temperature below which crude oil becomes plastic and will not flow. It is important to recovery and transport and is always

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determined. Pour points range from 32 °C to below −57 °C (90 °F to below −70 °F).

Basic Sediments and Water of the Crude oil

The Basic Sediment and Water content of crude oil is determined using small centrifuges that are driven by hand or by electric motor. A small measured volume of sample is diluted with solvent and placed in graduated glass containers that are then inserted into the centrifuge and spun for a few minutes at speeds of 2,000 to 4,000 rev/min.

Preparation of Oil Field Brine

Oil field brine was prepared by dissolving 30g of sodium chloride in 1000ml of distilled water to obtain a 0.5mol/dm^3 of the solution. This was to produce a stable crude oil emulsion having a constant salinity. The brine would be required in producing various mixtures of emulsion of crude oil. Salinity values range from 5% in September to about 17% in March in coastal region of Niger Delta. Most produced waters have between 8 – 20 percent salinity, hence a 0.5mol/dm^3 of solution was used for this investigation.

Preparation of Local Emulsifier

Palm kernel oil and cashew nut oil were used in the preparation of the local emulsifier. A mixture of the two oils would give an emulsifier that is soluble in oil and slightly soluble in water. Since the most problematic emulsion is water in oil, the use of water-soluble emulsion would be of great advantage. The addition of soap to the combination would make it highly soluble in water. Three blends of the local emulsifier were prepared using palm kernel oil and cashew nut oil in the following ratio, 40:40, 50:30, and 30:50 respectively.

Table 2.2 Percentage Composition of the Various Blends of Local Emulsifier

Materials	Diff. Blends of Local Demulsifier		
	Blend X_1 (%)	**Blend X_2(%)**	**Blend X_3(%)**
Palm Kernel Oil	40	50	30
Cashew Nut Oil	40	30	50
Liquid Soap	5	5	5
Starch	5	5	5
Calcium Hydroxide	2	2	2
Water	8	8	8

To formulate blend X_1, 120ml of palm kernel oil and 120ml of cashew nut oil were measured respectively with measuring and poured into a 500ml capacity beaker. The beaker containing the mixtures of the two oils was placed on a heated hot plate at a control temperature of 50^0 C. 8ml of water was mixed 5g of starch to form a viscous paste. The starch solution and 5ml of liquid soap were added to the mixture and gently stirred until a homogenous mixture was obtained. Finally, 2ml of calcium hydroxide solution was added to the mixture and stirred thoroughly. Blend X_2 and X_3 were obtained using the same procedure with different percent compositions. Each blend was stored in a bottle and label accordingly.

Treatment of Samples with Produced Local Demulsifier

100ml of crude oil emulsion from Y Field was filled into 5 measuring cylinders and labelled A, B, C, D and E. The 5 measuring cylinders were placed inside a water bath at a constant temperature of 40^0 C, the lease temperature. The locally prepared demulsifier was added to the 5 measuring cylinders in an increasing multiple amounts of 0.2 ml, beginning with 0.2 ml, 0.4 ml, 0.6 ml, 0/8 ml and 1.0 ml respectively and were allowed to settle for 30 minutes, 60 minutes and 90 minutes. The separation process was observed and recorded. The content of the 5 measuring cylinders was subjected to basic sediment and water separation process using a centrifuge machine and the results obtained were recorded. Following tests with the local demulsifiers, tests with foreign demulsifiers were conducted in order to replicate those done with locally produced materials. The purpose is to contrast the efficiency of demulsifiers prepared locally and those imported.

III. RESULTS ANALYSIS AND DISCUSSION

Results of Crude Oil Sample Properties

The following properties of the untreated crude oil emulsion of well K_2 of Y Field of Niger Delta were determined as outlined in chapter two and the results is presented in table 3.1 below.

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Table 3.1 Results of the Untreated Crude Oil Emulsion

S/No	Test properties of crude oil sample	Values before treatment
1	Density (g/ml)	0.884
2	Specific Gravity	0.89
3	API Gravity (^{0}API)	27.5
4	Pour Point (^{0} C)	9.3
5	Volume of Water (ml)	5.7
6	Sediment (%)	0.2

Results of Treated Emulsion with the diff. Blends of Demulsifiers

The basic sediment and water test was conducted on the crude oil emulsion after treatment with various volumes of the formulated demulsifiers, a pipette was inserted into the clean top dry oil in the measuring cylinder where the oil and water separation occurred and samples (5ml) were drawn into test tubes and topped with xylene to the 10ml mark. The tubes were then inserted into the centrifuge jack and spun for 10 minutes at 1000rpm. The centrifuge machine was allowed to come to a stop slowly and thereafter the result was observed and recorded. The results of the separation before centrifuging for each demulsifier was recorded and tabulated, and the results after centrifuging were determined and plotted as shown in figures 3.1, 3.2, 3.3 and 3.4 below for blend X_1, X_2, X_3 and PDMS respectively.

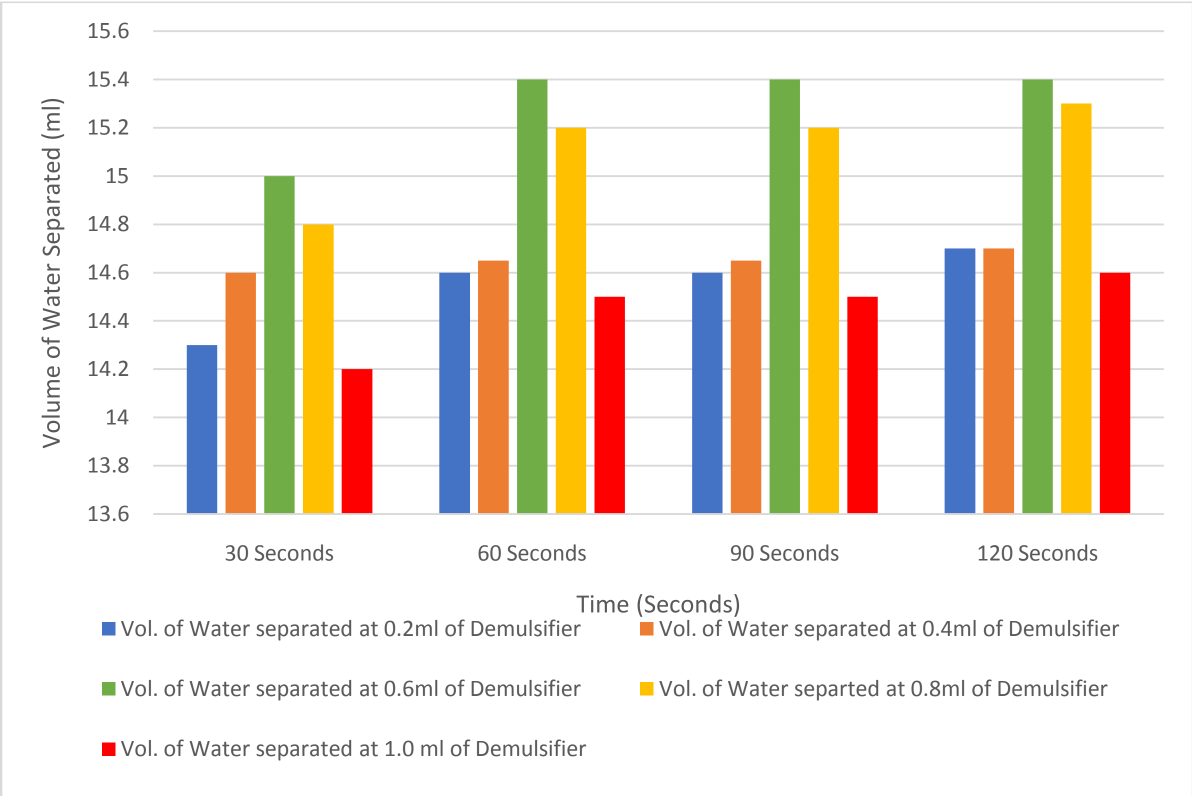

Figure 3.1 Water Separated versus Time for Blend X_1 Demulsifier

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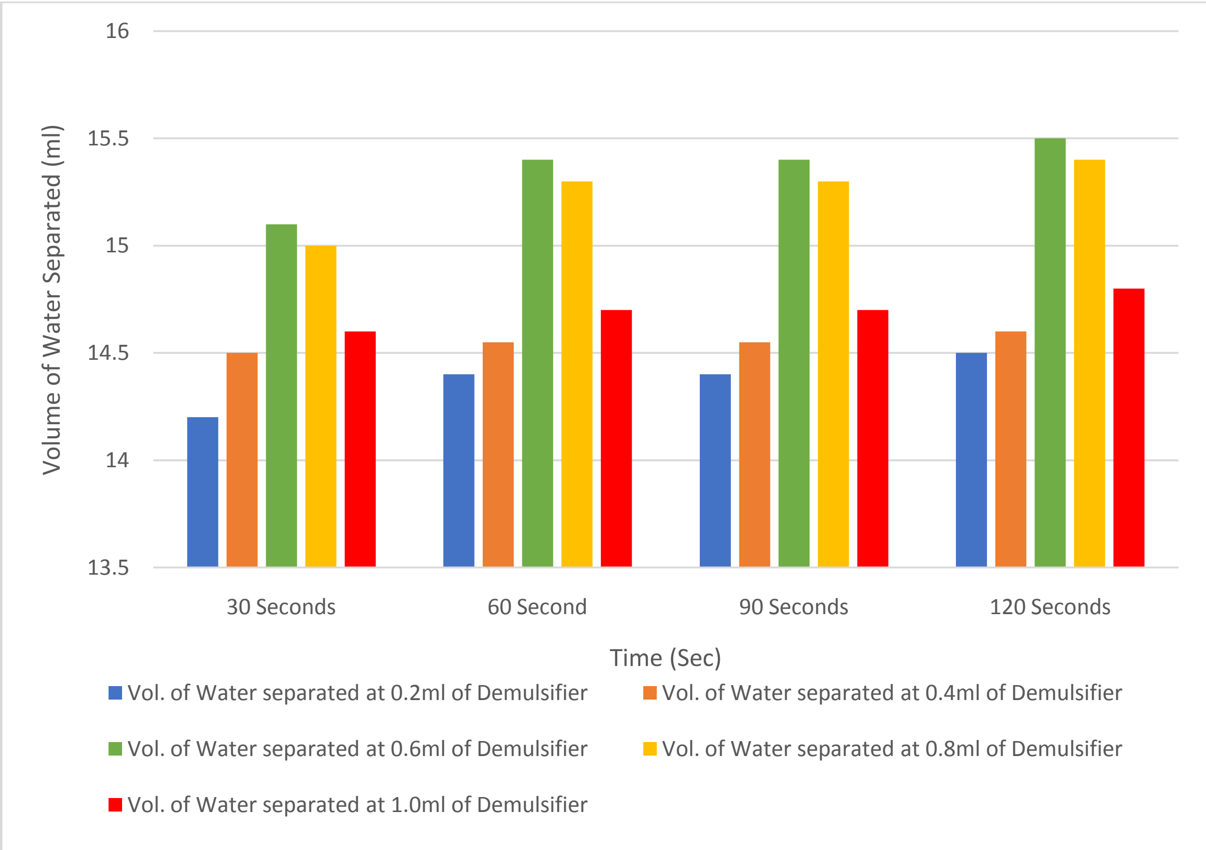

Figure 3.2 Water Separated versus Time for Blend X_2 Demulsifier

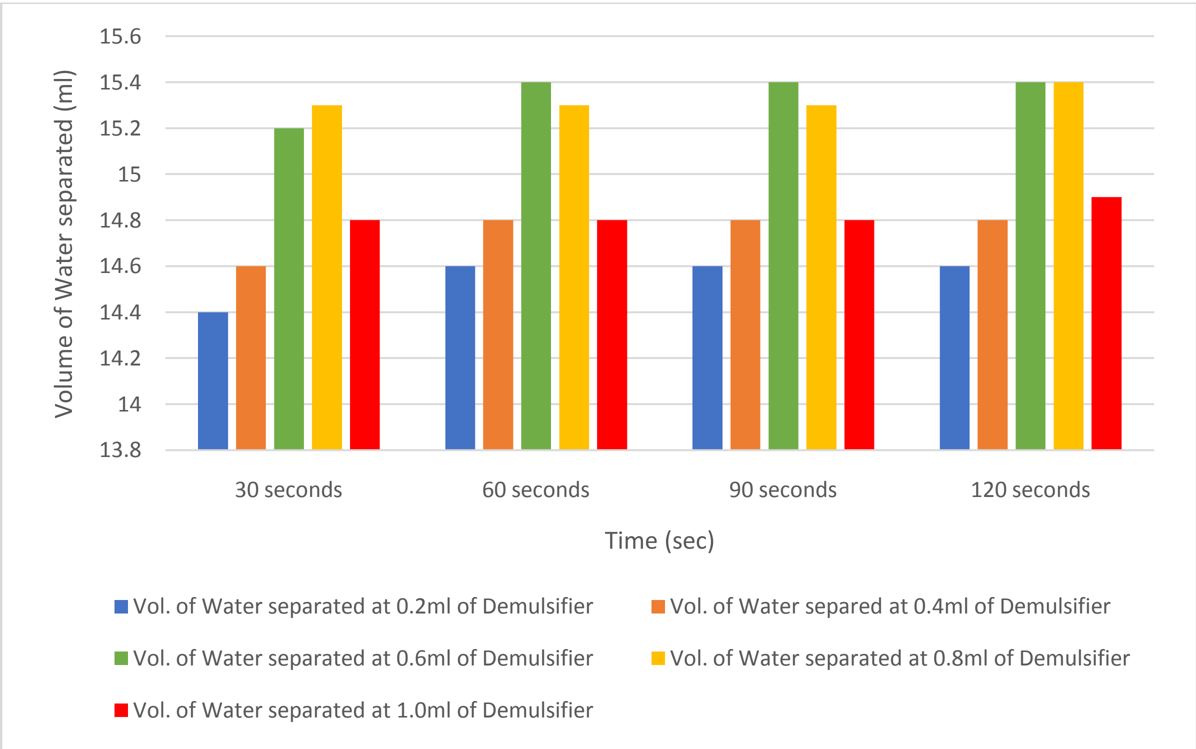

Figure 3.3 Water Separated versus Time for Blend X_3 Demulsifier

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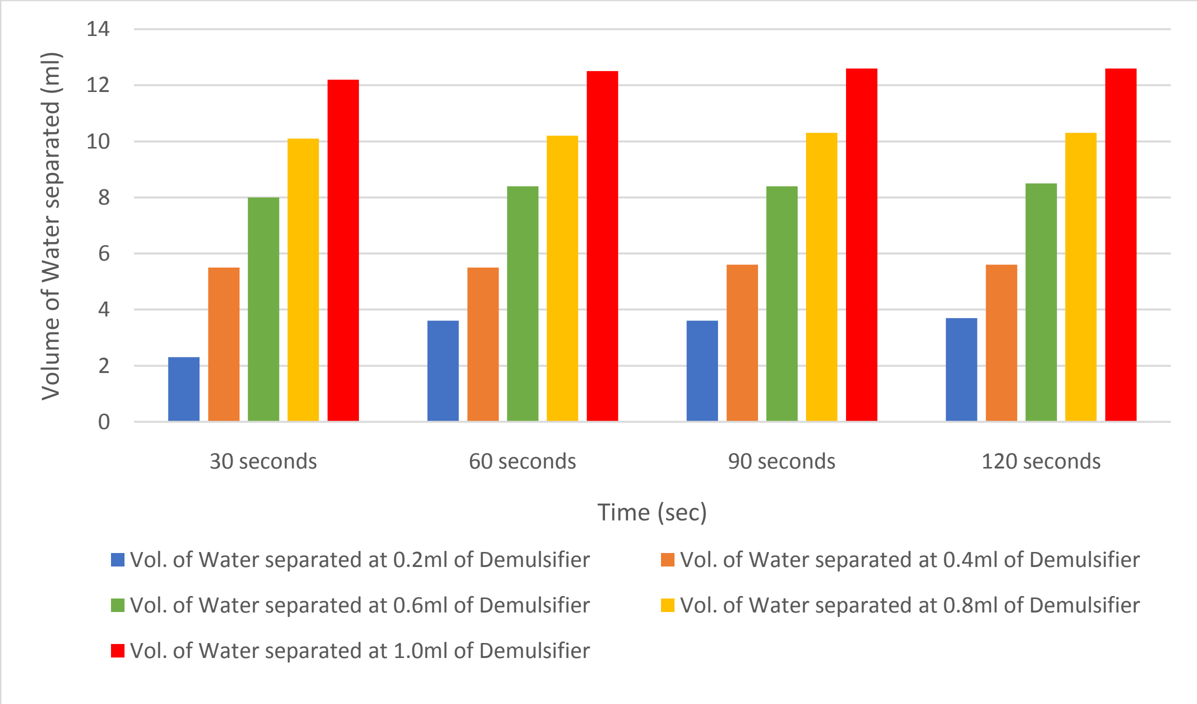

Figure 3.4 Water Separated versus Time for ForeignDemulsifier (PDMS)

Results Analysis of Crude Oil Properties after Treatment with the Prepared and Foreign Demulsifiers

There are significant differences in the initial values of the crude oil properties before and after treatment with the blended and foreign demulsifiers. Notably are the API Gravity, pour point and volume of water separated as shown in figure 3.4 below.

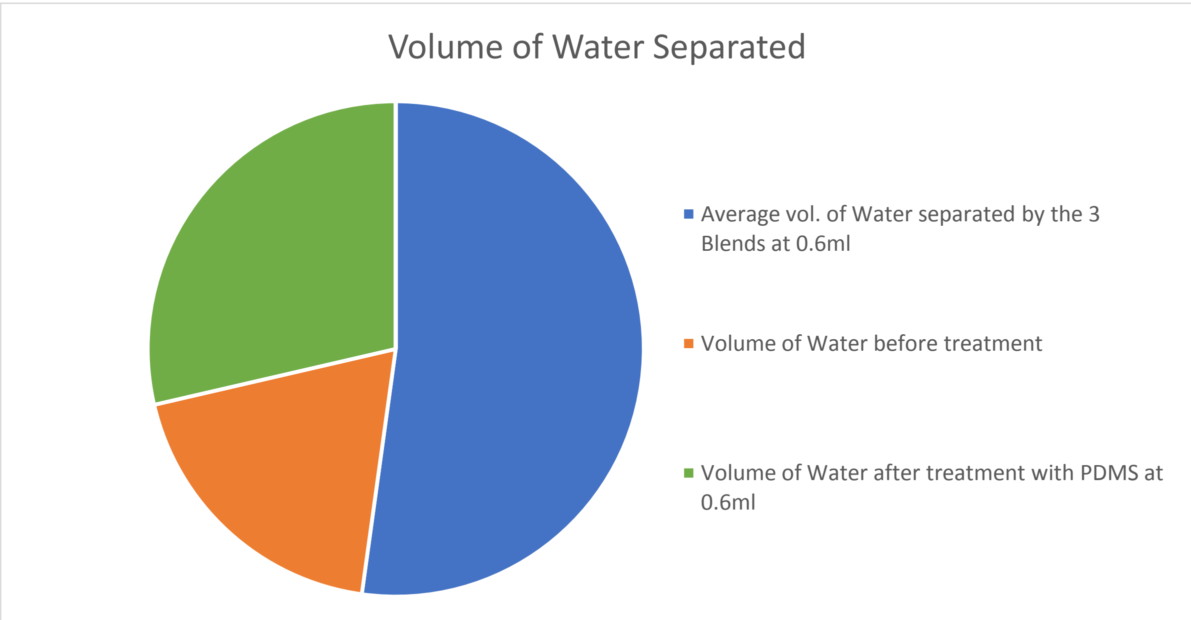

Figure 3.4 Volume of water separated before and after treatment.

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Results Analysis of Percentage of Basic Sediments and Water (BS & W) Left in the Treated Crude Oil

A maximum of 15.5ml resulting to 15.5% of water out of 100ml was released from the crude oil emulsion after treatment with 0.6ml dosages of the three blends of demulsifiers, while 8.5ml of water was released with the same dosage of polydimethylsiloxane (PDMS). However, at higher dosage of PDMS, more water was released. The quality of oil and water separated after demulsification with the various blends shows that all the blends produced good oil quality and water quality with sharp oil - water interface and complete separation within the time of 30mins to 120 minutes. The results of the basic sediment and water left in the treated crude oil with both the foreign and local demulsifier at the same and different dosages are presented in tables below

Table 3.2 Results of BS and W after treatment with the prepared and foreign demulsifier (PDMS) at 0.6 ml

BS & W	BlendX_1	BlendX_2	BlendX_3	PDMS
Oil recovered(%)	97.3	97.5	97.2	93.6
Water left (%)	2.7	2.5	2.8	6.4
Sediment (%)	-	-	-	-

Table 3.3 Results of BS and W after treatment with the prepared demulsifier at 0.6 ml and imported demulsifier (PDMS) at 1.4 ml

BS & W	BlendX_1	BlendX_2	BlendX_3	PDMS
Oil recovered (%)	97.3	97.5	97.2	97.6
Water left (%)	2.7	2.5	2.8	2.4
Sediment (%)	-	-	-	-

IV. CONCLUSIONS

After careful experimental tests and analysis were conducted on the crude oil emulsion sample, it was revealed that the locally blended demulsifiers proved highly effective. All the blends spontaneously resolved the emulsion in the crude oil sample producing nearly 100% dry crude oil with no trace of sediments. Three demulsifier blends X_1, X_2 and X_3 were prepared using palm kernel oil, cashew nut oil, liquid soap, locally prepared starch and Calcium Hydroxide. All three blends performed optimally at 0.6ml within 30 minutes in this work. The foreign demulsifier took 120 minutes at 1.0 ml to show similar effectiveness. Chemical demulsification of crude oil emulsion is best achieved when combined with other methods of treating emulsions like Heat and Gravity settling. Results also indicate that the blended local demulsifiers performed better than foreign demulsifiers.

A maximum of 15.5ml resulting to 15.5% of water was released from the crude oil emulsion. The quality of oil and water separated after demulsification with various blends shows that all the blends produced good oil quality and water quality with sharp oil - water interface and complete separation within the time of 30mins to 120 minutes. The results were in line with the specification allowable for the BS&W contained in crude oil for export and our local refineries as stipulated by the Nigerian Upstream Petroleum Regulatory Commission (NUPRC)

From the experimental findings and analysis above, it can be deduced that the quality of oil obtained after treatment with the various blends improved significantly, hence some locally formulated demulsifiers can be very effective in breaking out water from oil emulsion in less time than the foreign demulsifier. In these studies, all the three blends quickly resolved the emulsion in the crude oil sample with nearly 100% of dry crude oil. Some locally formulated demulsifiers can actually replace some foreign demulsifiers in resolving emulsification problems.

ACKNOWLEDGEMENT

We wish to thank the management of Federal university of Petroleum Resources, Effurun, Delta State for the conducive environment for carrying out the project work in no distant time.

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