

Optimum Comparison and Validation of Commercially Available Demulsifier and Locally Formulated Demulsifier for Crude Oil Emulsion Separation

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ABSTRACT

The need to develop a cost effective and efficient demulsifier in treating crude oil emulsions without compromising quality and environmental safety is a major concern to the oil industry worldwide. The production of crude oil along with water has been a primary problem in the petroleum industry. In this work, an emulsion breaking chemical (LFD) was formulated from locally sourced materials and experimentally compared with an imported commercially available demulsifier (sulphuric acid) to determine its effectiveness in crude emulsion resolution. Materials used are Castor oil, locally made liquid soap, Camphor, Alum, Cassava Starch and distilled water. It was tested on a crude oil emulsion sample from the Niger Delta field at a temperature of 85^of, under the same experimental condition with the imported commercially available demulsifier (sulphuric acid) as the control. The percentage volume of water separated by the locally formulated demulsifier (LFD) was 93.33% while the separated percentage volume of water by commercially available demulsifier (sulphuric acid) was 69.33%. This clearly showed that the locally formulated demulsifier had a better performance in terms of emulsion resolution than the commercially available imported demulsifier (sulphuric acid).

Keywords: Local materials, Coalescence, Demulsifier, Emulsion, Flocculation, Niger Delta.

1. Introduction

The most important objective of any oil production facility is the separation of water and other foreign materials from produced crude. Emulsions of oil and water are one of many problems directly associated with the petroleum industry, in both oil-field production and refinery environments, (R. Grace and Schramm, L.L. 1992). Whether these emulsions are created inadvertently or are unavoidable, as in the oil-field production area, or are deliberately induced, as in refinery desalting operations, the economic necessity to eliminate emulsions or

maximize oil-water separation is present. Emulsion problems in crude oil production and transportation requires expensive emulsion separation equipment such as water treaters, separators and coalescers. Hence, chemical demulsification is the most suitable method from both operational and economic point of view to break the crude oil emulsion (I.H. Auflem. 2002). Among chemical agents, interfacial-active demulsifiers, which weaken the stabilizing films to enhance droplets coalescence, are preferred due to lower addition rates needed. However, these demulsifiers are costly and pose significant threat to the environment. It becomes imperative to develop cheap and environmentally friendly demulsifiers from locally source raw material.

1.1 Background of Study

Emulsion is defined as a system in which one liquid is relatively distributed or dispersed, in the form of droplets, in another substantially immiscible liquid. Emulsions have long been of great practical interest due to their widespread occurrence in everyday life which occurs due to reliance of the behaviour of the emulsion on the magnitude and range of the surface interaction. They may be found in important areas such as food, cosmetics, pulp and paper, biological fluids, pharmaceutical, agricultural industry, and petroleum engineering. In production and flow assurance, the two commonly encountered emulsion types are water droplet dispersed in the oil phase and termed as water-in-oil emulsion (W/O) and if the oil is the dispersed phase, it is termed oil-in-water (O/W) emulsion (D. Langevin, S. Poteau, I. H'énaut, and J. F. Argillier, 2004). When there is dispersion (droplets) of one liquid in another immiscible liquid is called emulsion. The phase that is present in the form of droplets is the dispersed or internal phase, and the phase in which the droplets are suspended is called the continuous or external phase. For produced oilfield emulsions, one of the liquids is aqueous and the other is crude oil. The amount of water that emulsifies with crude oil varies widely from facility to facility. It can be less than 1% and sometimes greater than 80%.

In a true emulsion, either the drop size must be small enough that forces from thermal collisions with molecules of the continuous phase produce Brownian motion that prevents settling, or the characteristics of the interfacial surfaces must be modified by surfactants, suspended solids, or another semisoluble material that renders the surface free energy low enough to preclude its acting as a driving force for coalescence.

1.1.2 Characteristics of Emulsion

Crude oil emulsions are complex and should be characterized as completely as possible. Droplet-size distribution, interfacial phenomena, and the nature of organic and inorganic components are important. The viscosity of the emulsion is affected by both the water content and droplet size distribution [S. D. Taylor, 1988, D. G. Thompson, A. S. Taylor, and D. E. Graham, 1987]. The increase in aqueous phase of the emulsion leads to an increase in viscosity of emulsion which in turn aggravates flow of emulsion in conduct either at the sand face or through the surface facilities [R. Espinoza and W. Kleinitz, 2003, K. Sunil, A. Abdullah, and N. S. Meeranpillal, 2007]. Stable water-in-oil emulsions have been generally found to exhibit high interfacial viscosity and/or elasticity modulus. Viscosity of crude oil emulsion was found to increase with increase in water and decreased with increase in speed of rotation of spindle when demulsifier is added [N. H. Abdurahman and W. K. Mahmood, 2012].

An emulsion's characteristics change continually from the time of formation to the instant of complete resolution. Accordingly, aged emulsions can exhibit very different characteristics from those that fresh samples do. This is because any given oil contains many types of adsorbable materials and because the adsorption rate

of the emulsifier and its persistence at the interface can vary. The emulsion characteristics also change when the liquid is subjected to changes in the following:

- Temperature
- Pressure
- Degree of agitation

1.1.3 Appearance and Color

Color and appearance is an easy way to characterize an emulsion. The characterization becomes somewhat easy if the emulsion is transferred into a conical glass centrifuge tube. The color of the emulsion can vary widely depending on:

- Oil/water content
- Characteristics of the oil and water

The common colors of emulsions are dark reddish brown, gray, or blackish brown; however, any color can occur depending on the type of oil and water at a particular facility. Emulsion brightness is sometimes used to characterize an emulsion. An emulsion generally looks murky and opaque because of light scattering at the oil/water interface. When an emulsion has small diameter droplets (large surface area), it has a light color. When an emulsion has large diameter droplets (low total interfacial surface area), it generally looks dark and less bright. Understanding the characteristics of an emulsion by visual observation is an art that improves with experience.

1.1.4 Formation of Emulsions

Crude oil emulsions form when oil and water (brine) come into contact with each other, when there is sufficient mixing, and when an emulsifying agent or emulsifier is present. The amount of mixing and the presence of emulsifier are critical for the formation of an emulsion. During crude oil production, there are several sources of mixing, often referred to as the amount of shear, including: Flow through reservoir rock, Bottomhole perforations/pump, Flow through tubing, flow lines, and production headers, Valves, fittings, and chokes, Surface equipment, Gas bubbles released because of phase change. The amount of mixing depends on several factors and is difficult to avoid. In general, the greater the mixing, the smaller the droplets of water dispersed in the oil and the tighter the emulsion. Emulsion studies have shown that the water droplets can vary in size from less than 1 μm to more than 1000 μm . The second factor important in emulsion formation is the presence of an emulsifier. The presence, amount, and nature of the emulsifier determines, to a large extent, the type and "tightness" of an emulsion. The natural emulsifiers in crude are resident in the heavy fraction. Because there are different types of crudes and because these crudes have different amounts of heavy components, the emulsifying tendencies vary widely. Crude with a small amount of emulsifier forms a less stable emulsion and separates relatively easily. Other crudes contain the right type and amount of emulsifier, which lead to very stable or tight emulsions. Emulsions may be encountered at all stages in the petroleum recovery and processing industry (drilling fluid, production, process plant, and transportation emulsions)

1.1.5 Emulsion Classification

Emulsion viscosity can be substantially greater than the viscosity of either the oil or the water because emulsions show non-Newtonian behavior. This behavior is a result of droplet crowding or structural viscosity. A fluid is

considered non-Newtonian when its viscosity is a function of shear rate. A closely related and very important property, especially for demulsification, is the interfacial viscosity, or the viscosity of the fluid at the oil/water interface. As mentioned water-in-oil emulsions form rigid interfacial films encapsulating the water droplets. These interfacial films stabilize an emulsion by lowering IFT and increasing interfacial viscosity. These films retard the rate of oil-film drainage during the coalescence of water droplets, thereby greatly reducing the rate of emulsion breakdown. The oil-drainage rate depends on the interfacial shear viscosity. High interfacial viscosities significantly slow the liquid drainage rate and thus have a stabilizing effect on the emulsion. Emulsion interfacial viscosity plays a very important role in demulsification.

Produced oilfield emulsions can be classified into three broad groups:

- Water-in-oil
- Oil-in-water
- Multiple or complex emulsions

Water-in-oil emulsions consist of water droplets in a continuous oil phase, and oil-in-water emulsions consist of oil droplets in a water-continuous phase. **Figs. 1 and 2** show the two basic (water-in-oil and oil-in-water) types of emulsions. In the oil industry, water-in-oil emulsions are more common (most produced oilfield emulsions are of this kind); therefore, the oil-in-water emulsions are sometimes referred to as "reverse" emulsions.

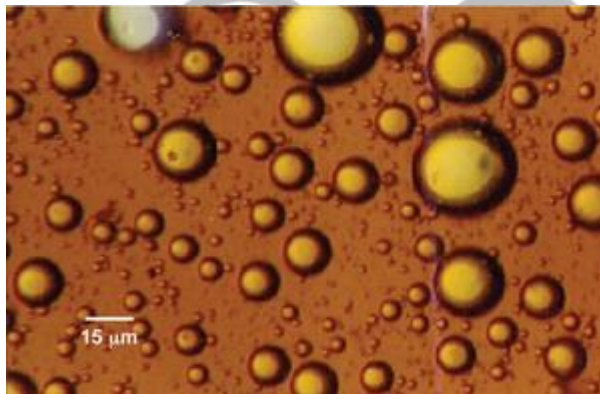


Fig. 1.1 – Photomicrograph of water-in-oil emulsion.

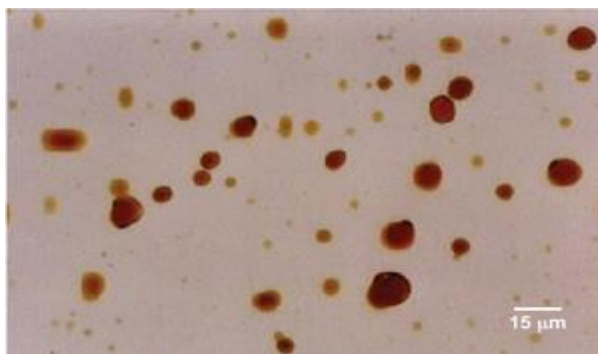


Fig. 1.2 – Photomicrograph of oil-in-water emulsion.

Multiple emulsions are more complex and consist of tiny droplets suspended in bigger droplets that are suspended in a continuous phase. For example, a water-in-oil-in-water emulsion consists of water droplets suspended in larger oil droplets that, in turn, are suspended in a continuous water phase. **Fig. 3** shows an example of a multiple emulsion.

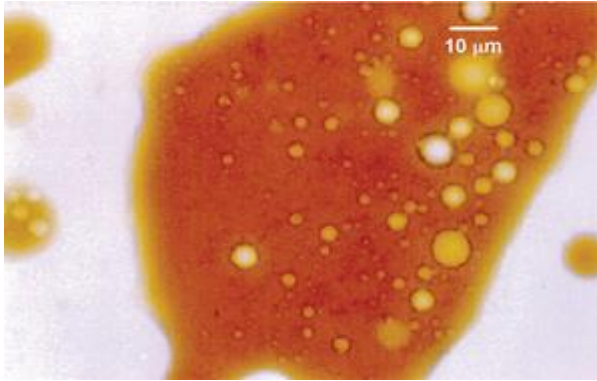


Fig. 1.3 – Photomicrograph of water-in-oil-in-water emulsion.

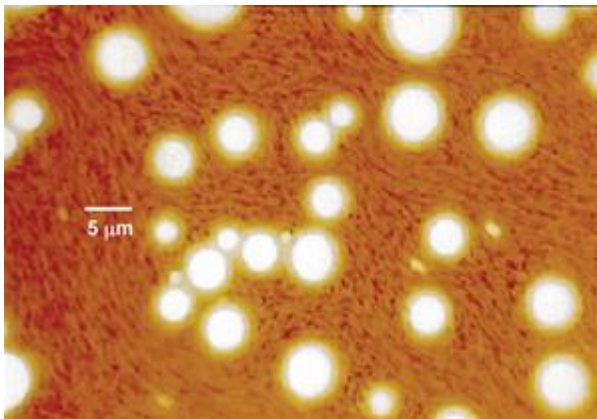


Fig. 1.4 – Photomicrograph of emulsion showing the presence of solids.

Water-in-oil crude oil emulsions may be encountered at all stages in the petroleum production and in processing industry. With presence of water, they are typically undesirable and can result in high pumping costs and pipeline corruptions and increase the cost of transportation. Reduced throughput is needed to introduce special handling equipment, contribute to plugging of gravel pack at the sand phase, and affect oil spill cleanup.

1.2 Objectives of Study

This research is focused primarily to formulate local demulsifier from locally sourced raw materials and apply the formulated local demulsifier on water-in-oil emulsion for demulsification.

2.1 Demulsification process

A number of general rules help to form the basic philosophy of how emulsion behave within commercial emulsion breaking (Grace, 1992). Firstly, petroleum emulsions are composed primarily of immiscible liquids.

Separation should be the natural tendency of these liquids, by providing the density different between the liquid that are exists. Secondly, the gravitational settling rate is dependent on the surface tension of the droplets that form the internal phase of the emulsion. Large droplets have less surface tension as a function of mass than small droplets.

Therefore, anything that can be done to increase the droplets size or coalescence will increase the rate of separation. Thirdly, an emulsion is stable within a given environment. Varying the environment may affect the stability of an emulsion and allow the phases separation. Finally, a stable emulsion exists only when emulsifying agents are present. Neutralization, alteration or elimination of the emulsifying agents will allow immiscible liquids to separate.

From the above four generalizations it becomes noticeable that a number of options exist in emulsion breaking. Any single change in these areas may result in the resolution of an emulsion.

2.2 Demulsifier formulation

Demulsifiers can be used singularly or in combinations of two or more.

Finding a demulsifier system that works well is often done by trial and error. Grace (1992) stated that the selection process has historically been viewed as a “black art”, which produces as many failures as successes. But the failures can be eliminated with the increasing understanding of emulsions and emulsion-breaking chemicals, the development of new test procedures and devices, and well-organized method of chemical selection. The best demulsifiers are one that can reduce the interfacial shear viscosity, increases the interfacial mobility and destabilizing the water-in-oil emulsion.

2.3 Demulsifiers performance

Different types of demulsifiers will give the different way in demulsification process. Knowledge of formation and stability of crude oil emulsions, types of demulsifiers, demulsification mechanisms and so on are very important since it can be useful in the demulsification process of crude oil emulsions. Thus, it is frequently observed in studies of parameters that can affect the formation of stability of the crude oil emulsion.

3.0 Materials and Methods

3.1 Materials

The sample of crude oil emulsion utilized in this research was gotten from an oil field in Niger Delta region of Nigeria. Two Demulsifiers, locally formulated demulsifier (LFD) and a commercially available imported (H₂SO₄) were used in treating this emulsion. The centrifugal agitation method was used to determine the Demulsifier most effective in breaking the emulsion. The materials used in formulating the demulsifier are shown in Table 1 below.

Content	Function	Weight/Vol
Alum	To facilitate settling of sediments.	5g
Castor Oil	It acts as the lipophilic agent in the demulsifier.	30ml
Starch	Coalescing of the tiny water droplets.	5g
Liquid Soap	Destabilization of emulsion film	50ml
Camphor	It improves the demulsifier performance	10ml

Table 1: Materials Used for Locally Formulated Demulsifier (LFD)

Apparatus for the experiment include weighing balance, measuring cylinder (50ml graduated), beaker,

magnetic heat- stirring machine, filter paper, measuring cylinder (200ml graduated), Hamilton beach mixer, stop watch, syringe, micro- pipette, thermometer, centrifuge.

3.2 Methods

Water-in-oil emulsion was formed in the laboratory as follows:

- 3.2.1 180ml of crude oil was blended together with (20ml) of liquid surfactant in a beaker for emulsification. The crude oil/surfactant blend was placed in a Hamilton beach mixer for agitation.
- 3.2.2 Water of 20ml representing 10% of the emulsion was then gradually added to the oil phase at a consistent rate of agitation. Agitation continued until the emulsion reached ambient temperature without forced cooling.



Figure 1: Hamilton Beach Mixer

Locally formulated demulsifier (LFD) procedure are as follows:

- 3.2.2.1 A solution of 10g of camphor dissolved in 30ml of castor oil was stirred and heated (150°F) in a Magnetic heating and stirring machine until homogeneity of solution is achieved.
- 3.2.2.2 A mixture of 5g of starch (from cassava flour), 50ml of detergent, and 5ml of Alum solution was prepared in another beaker and put into the camphor castor oil solution.
- 3.2.2.3 All precipitates or sediments were filtered off after the new mix or blend above has been further stirred and heated for 2 hours.

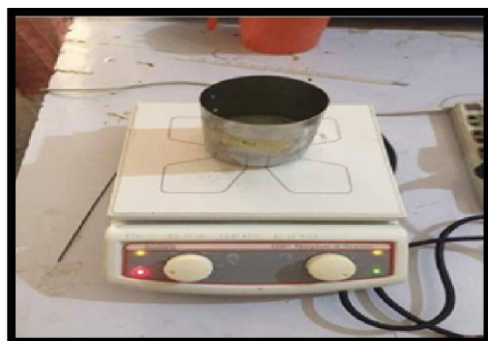


Figure 2: Magnetic Heat-Stirring Machine

2.2.1. Analytical Procedure

The first experiment was done taking sulphuric acid (H₂SO₄) to be its demulsifying agent while the second was carried out using locally formulated demulsifier (LFD).

- A 75ml of emulsified crude oil were poured into each of the 12 measuring tubes. All measuring tubes were labeled according to the concentration of Demulsifier to be used for the analysis. Six bottles of demulsifier were utilized with concentrations of 0.2 milli litre, 0.4 milli litre, 0.6 milli litre, 0.8 milli litre,

1.0 milli litre, and 1.2 milli litre of demulsifier.

- All the cylinders were manually agitated properly to ensure fluid mobility
- All tubes containing samples with varying dose of demulsifier were placed into a centrifuge for agitation to achieve proper mixture.
- The revolutions in one minute (rpm) were noted down.
- After the time of rotation had passed, the test tubes placed in the centrifuge were measured and also the separation of the oil from the water recorded
- The procedure was done again for the entire samples and the results were also recorded.



Figure 3: Centrifuge

4.0 Results and Discussion

The results of the emulsion resolution using the two demulsifiers (a locally formulated Demulsifier (LFD) and commercially available Demulsifier (H₂SO₄)) at a constant treatment temperature of 85oF and time interval of 2 to 12minutes and at a varying RPM of 1000 and 1500 summarized in Table 2,3.1,3.2,3.3,3.4 and 3.5 accordingly.

Crude Properties	
Density	0.864g/Cm ³
Api	32.46 ^o
Viscosity	2.9 Cp

Table 2: Characteristics of Crude Oil Utilized

Vol. of Demulsifiers(ml)	Separated Water Volume(ml)					
	2min.	4min.	6min.	8min.	10min.	12min.
0	0	0	0	0	0	0

Table 3: Emulsion without Demulsifier at a Speed of 1000 Rpm

Vol. of Demulsifier (MI)	Separated Water Volume (MI)					
	2min.	4min.	6min.	8min.	10min.	12min.
0.2	0.5	0.7	0.8	1.0	1.1	1.2
0.4	0.9	1.1	1.3	1.5	2.0	2.2
0.6	2.2	2.5	2.5	2.7	2.8	2.8
0.8	2.9	3.0	3.2	3.2	3.2	3.3
1.0	3.0	3.1	3.3	3.5	3.5	3.6
1.2	3.6	3.7	3.9	4.0	4.0	4.1

Table 4: Emulsion Treatment with Sulfuric Acid at 1000 Rpm and 85 °F

Percentage volume of water separated using H₂SO₄ at 1000 rpm

$$= \frac{\text{Vol. water separated}}{\text{Total vol. of water in the emulsion 1}} \times 100$$

Total vol. of water in the emulsion 1



$$= \frac{4.1}{7.5} \times \frac{100}{1} = 54.67\%$$

Vol. of Demulsifier(ml)	Separated Water Volume (ml)					
	2min.	4min.	6min.	8min.	10min.	12min.
0.2	0.6	0.9	1.3	1.5	1.6	1.8
0.4	1.0	1.4	1.5	1.8	2.2	2.4
0.6	2.3	2.6	2.9	2.9	3.1	3.2
0.8	3.0	3.3	3.6	3.8	3.9	3.9
1.0	3.9	4.3	4.9	5.1	5.1	5.3
1.2	4.2	4.8	4.9	5.2	5.3	5.5

Table 5: Emulsion Treatment with LFD at 1000 Rpm and 85of

Percentage volume of water separated using LFD at 1000 rpm.

$$= \frac{\text{Vol. water separated}}{\text{Total vol. of water in the emulsion}} \times 100$$

$$= \frac{11.1}{15.5} \times 100 = 71.61\%$$

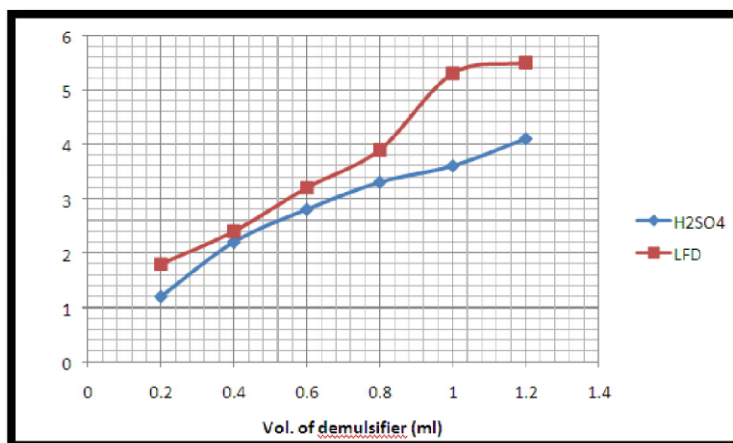


Figure 4: Volume of Separated Water Vs Volume of Demulsifier (Ml) at 85of and 1000 Rpm.

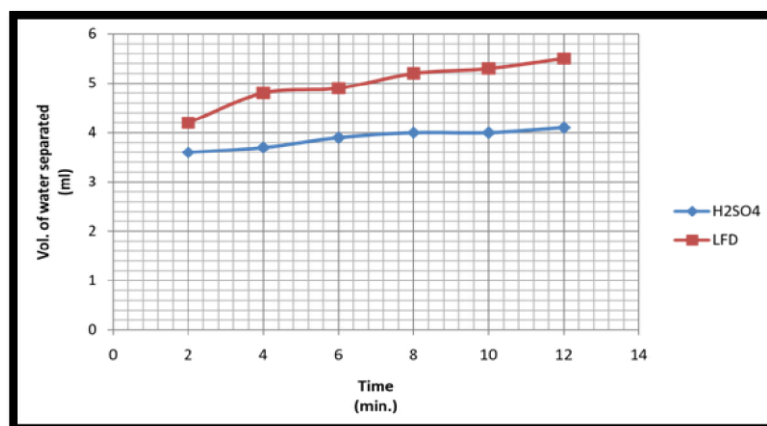


Figure 5: Volume of Separated Water (Ml) Vs Time (Min.)@ 1000 Rpm

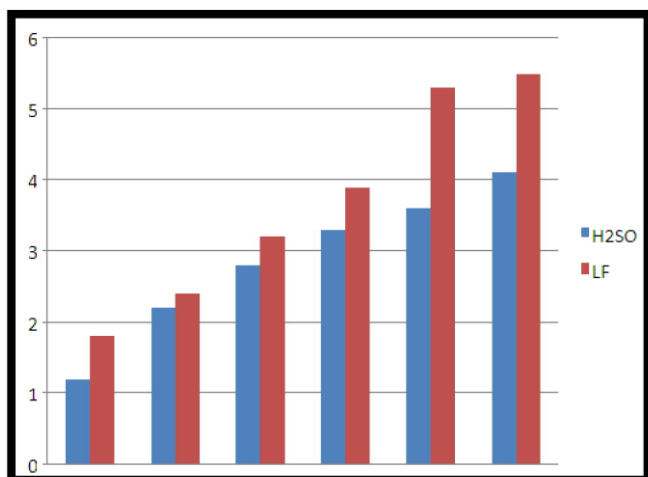


Figure 6: A Bar Charts Representing the Performance of H2SO4 and LFD at 1000 Rpm

Vol. of Demulsifier(ml)	Separated Water Volume(ml)					
	2min.	4min.	6min.	8min.	10min.	12min.
0.2	0.5	0.8	0.9	1.0	1.2	1.3
0.4	1.0	1.1	1.4	1.6	1.7	2.3
0.6	2.3	2.5	2.6	2.8	2.9	2.9
0.8	2.9	3.0	3.3	3.3	3.4	3.4
1.0	3.1	3.2	3.4	3.5	3.6	3.7
1.2	4.2	4.5	4.9	5.1	5.2	5.2

Table 6: Emulsion Treatment with H2SO4 at 1500 Rpm and 85°f

Percentage volume of water separated using H2SO4 at 1500 rpm

$$= \frac{\text{Vol. water separated}}{100 \text{ Total vol. of water in the emulsion}} \times 100$$

$$= \frac{5.2}{7.5} \times 100 = 69.33\%$$

Vol. of Demulsifier(Ml)	Separated Water Volume(Ml)					
	2min.	4min.	6min.	8min.	10min.	12min.
0.2	0.8	1.2	1.5	1.8	2.0	2.3
0.4	1.5	1.9	2.5	3.0	3.3	3.8
0.6	3.0	3.8	4.0	4.2	4.3	4.3
0.8	3.9	4.2	4.5	4.6	4.6	4.7
1.0	4.5	4.9	5.0	5.2	5.5	6.9
1.2	5.1	5.3	5.8	5.9	6.4	7.0

Table 7: Emulsion Treatment with LFD at 1500 Rpm and 85°f

Percentage volume of water separated using LFD at 1500 RPM.

$$= \frac{\text{Vol. water separated}}{\text{Total vol. of water in the emulsion}} \times 100$$

1

$$\frac{7.0 \times 100}{7.5}$$

$$1 = 93.33\%$$

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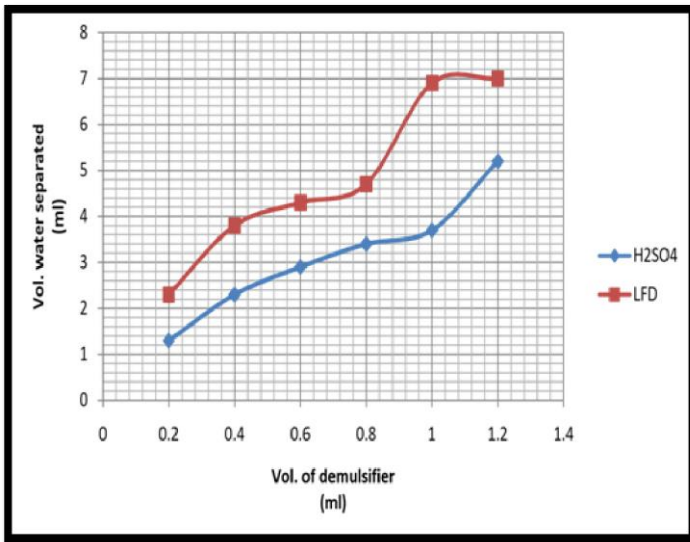


Figure 7: Vol. of Separated Water Vs Vol. Demulsifier (ml) @ 1500 Rpm and 85^of

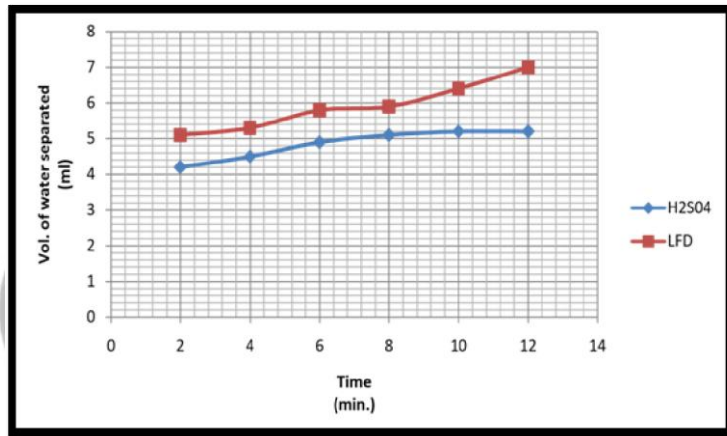


Figure 8: Volume of Water Separated (ml) Vs Time (Min) @ 1500 Rpm and 85^of

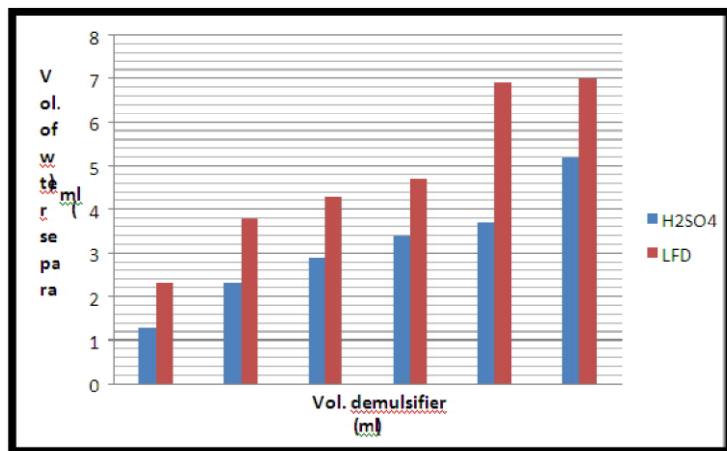


Figure 9: A Bar Chart Representing the Performance of H₂so₄ and Lfd @ 1500 Rpm and 85^of



Figure 10: The Schematic Representation of Resolved Emulsion

From Table 3 the experiment was done without a Demulsifier and there was no separated water from the emulsion which shows that no amount of agitation will break an emulsion without a Demulsifier. At a speed of 1000 rpm at 85oF better results were obtained using LFD as shown in Tables 3.3 and Fig 3.3. Percentage of water separated was 73.33%. At a speed of 1500 rpm at 85oF as displayed in Table 7 and Fig 3.5. The amount of water in percentage separated was 93.33%. All the samples were agitated with centrifuge for 12minutes in each case. This shows that locally formulated demulsifiers (LFD) were more effective in emulsion breaking than imported sulphuric acid (H₂SO₄)

5.0 Conclusion

In oil field and water treatment operations, producer of crude oil is interested in three parts of demulsification. Rate or speed at which the detachment occurs, quantity of water remaining in the crude oil after separation, and quality of the detached water for disposal. In this work, a demulsifier was formulated from locally sourced material (LFD). It was tested on a crude oil emulsion sample from the Niger Delta's field in Nigeria at a temperature of 85oF in comparison with a commercially available imported demulsifier (sulfuric acid). The separated water percentage by volume using the locally formulated demulsifier (LFD) was 93.33%, while the separated water using foreign demulsifier (Sulfuric acid) was 69.33%. The LFD also had a faster rate of separation and a better quality of water separated than the commercially available imported demulsifier (sulfuric acid). The results clearly demonstrated that cheap local chemicals with demulsification properties can be used to successfully break emulsions and enhance the quality of produced crude oil. The locally formulated demulsifier (LFD) is not corrosive and does not

contain organic chloride, bromides, or lead, hence will not cause any refining problems

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