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Demulsification of Water-In-Oil Emulsions with Locally Formulated Emulsion Breaker for Production Operations

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ABSTRACT

Over the year, emulsification has been a prevailing challenge in oil and gas production operations however, there have been commensurate solutions in the demulsification of water from crude. Interestingly, demulsification has been achieved in the field with the aid of imported demulsifiers which of course has caused adverse effects on Nigeria's economy. Meanwhile, most raw materials can be sourced locally in-country, therefore, there is a need to harness the local materials to reduce or stop capital flights associated with the importation of oilfield chemicals. In this study, a locally formulated demulsifier was used for demulsification exercise on crude oil samples obtained from Lekoil field, and terminal in the Andoni Ikuru town area of Niger Delta, Nigeria. The bottle-test method was adopted for the separation of water from crude oil emulsions and its efficacy was ascertained at temperatures of 20°C, 40°C, and 60°C upon investigated demulsifiers concentration of 0 %, 1 %, 5 % and10 % respectively. The locally formulated demulsifier was compared with a commercial demulsifier(PHASETREAT). Thus, areas of focus were on the physical properties of both demulsifiers and crude oil samples which include pH, viscosity, specific gravity, density, API gravity as well as the rate of separation obtained. Experimental results showed that the locally formulated demulsfier performed better when compared with the commercial demulsifier, and it is also environmentally friendly. Therefore, the locally sourced demulsifier can be substituted for the commercial demulsifier. This experiment provides an opportunity for the advancement of the local content drive.

KEYWORDS: Salinity, demulsifier, temperature, specific gravity, density

I. INTRODUCTION

Water-in-oil emulsions stability is always encountered at many stages during the production and processing of crude oils. These emulsions formation is always or generally caused by the presence of resins and asphaltenes which play the main role of "natural emulsifiers", and are also caused by wax and solids that accompany the crude. These components can organize and form rigid films at the oil/water interface Effective separation of oil and water is essential to ensure the crude oil quality and the low cost of oil production. Chemical demulsification forms the most important step in breaking water-in-crude oil emulsions. Various techniques are used to break these emulsions, among which the most widely used consists of adding small amounts of demulsifiers.

[1]. These surface-active molecules adsorb at the oil-water interface and accelerate the phase separation.

Among the most critical aspects of petroleum production no matter what the production system is, separating produced crude from water and basic sediments. The most important objective of any oil production facility is the separation of water and other foreign materials from the produced crude. [2]. The breaking of these crude oil and water emulsions constitutes one of the challenging problems in today's petroleum industry. Oftentimes, crude oil production contains water as the main unavoidable associate or by-product thus, nearly 90% of crude contains oil/water emulsions. The gradual entry of water into oil-bearing formations and the arrival of secondary and tertiary recovery methods have led to the development of new technologies that can be used to break crude oil and water emulsions. Following a series of investigations and confidence results in the breaking of water-in-petroleum emulsions is not yet completely understood, particularly as far as the added chemical demulsifiers role is concerned, and much research is still required Hence Demulsifiers performance has to therefore be improved from the application as well as from the environmental point of view. Recently made formulations must be less toxic and at least as efficient as classical chemical families. Authors confirmed that crude oil is most often produced as a water-in-oil emulsion and the water must be removed (down to a level of <0.5%), in a process that is usually called demulsification or dehydration, which consists of forcing the coalescence of water droplets and producing their separation by settling. In order to properly separate the water from the water-in-oil emulsion, demulsifiers are used as process aids. The processes involved in the breaking of these emulsions are carried out by using synthetic surfactants (Demulsifiers) which are added to the water-in-oilemulsion. The function of the demulsifiers consists of the demulsification and prevention of re-emulsification by breaking the protective film which is formed on the surface of water drops, by the emulsifying agent Separation of the water-in-crude emulsion is a technically big challenge in the petroleum industry Making use of an effective demulsifier in demulsification process can save millions of Naira every year in operation costs.

Table 2 shows the materials for local demulsifier production and their importance while table 1 shows the equipment and their functions.

[3]. An increase in water content in oil of one percent canresult in transportation costs increasing by three to five percent (3-5%) for each transfer and the use of a demulsifier can also reduce oilfield corrosion.

The research work aimed at demulsifying emulsified crude oil with locally formulated demulsifier and the main objectives is to characterize the physical properties of the crude oil samples, commercial and locally sourced demulsifiers, evaluate the rate of separation with respect to concentration,n and determine the rate of separation as a function of temperature.

RESEARCH ACTIVITY

STUDY AREA

Crude oil samples for experimental analyses were obtained from Lekoil field, and terminal in the Andoni Ikuru town area of Niger Delta, Nigeria. Representative sampling obtained from the top and bottom of the tank in a loose and completely covers d plastic can and samples were preserved at ambient temperature of 5^oC.

MATERIALS AND EQUIPMENT

The materials and equipment used for this study are summarized in tables 1 and 2.

Table 1: List of Equipment

Item	Equipment/Apparatus	Type/Model	Function
1	Digital Electronic Viscometer	Drawell HBVD-2-320- 	Used to determine highly viscous fluids at speed of 320000000mPaS
2	Pycnometer	290 Cromocol 	Used to determine fluid density

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3	Hydrometer	Vee Gee Scientific 67511H 	determining the specific gravity of a liquid
4	Centrifuge machine	. Pyrex, England 	To separate components of a fluid.
5	Water bath	WB-12 	Constant temperature equipment, providing heat source for varieties of devices that need heating.

Table 2: Materials for local demulsifier production and their importance

S/N	Materials	Source	Location	Importance
1	Olive Oil	Mixture of fatty acid glycerides and non-glycerides	Port Harcourt	Edible cream oil that acted as solvent for oil camphor and also increased the lipophilic properties of the crude oil
2	Camphor powder	Conifer/Pinene tree (Turpene)	Port Harcourt	This formed the lipophilic end of the local demulsifier produced
3	Shear Butter	Extract from the nut of the African shea tree		This served as the bulking agent in the locally formulated produced
4	Starch	Cassava	Port Harcourt	This formed the hydrophilic end of the locally Produced demulsifier because of its strong affinity for water
5	Distilled water	Steam	Port Harcourt	This was used as solvent for the starch solution
6	Liquid Soap	Saponification of	Port Harcourt	This served as the binder

	fatty acids and alkaline	for the demulsifier produced from locally sourced materials to bind the lipophilic and the hydrophilic end

PRODUCTION OF LOCAL STARCH FROM CASSAVA

Some quantity of dry cassava starch was purchased from a local market around at mile three in Port Harcourt. 300 g of the dry cassava starch was dissolved in 300 g of cold distilled water to form a solution. Again, some quantity of distilled water was made to boil on a gas burner at 212 ^{0}F. 310.9 g of the boiled distilled water was added to the starch solution and gently stirred to form a paste- like solution.

PRODUCTION OF LOCAL DEMULSIFIER

To formulate blend 1, 10g of the synthetic powder camphor was measured and poured into a beaker. Containing 30 g olive oil, placed on a heated hot plate at a regulated temperature of 40°C gently stirred to dissolve the camphor. 10g of paraffin wax was then added to the hot camphorated Olive oil. Next, 30 g of prepared cassava starch added to the mixture while stirring still continued. Finally, 20g of the prepared liquid soap was added to the entire mixture and stirred gently for 60 minutes at 40 °C on the heated hot plate to obtain a homogenous blend of the local demulsifier. To formulate blends 2, 3 and 4, the same procedure used for the formulation of blend 1 was employed

CHARACTERIZATION OF PHYSICAL PROPERTIES OF CRUDE OIL SAMPLES, COMMERCIAL AND LOCALLY SOURCED DEMULSIFIERS

DETERMINATION OF VISCOSITY OF OLIVE OIL

Digital electronic viscometer was used for the viscosity test. The digital electronic viscometer measures Fluid viscosity at a given share rate. Viscosity is a measure of the fluid's resistance to flow. The principle of operation of the digital electronic viscometer is to rotate a spindle (which is immersed in the test fluid). Through a calibrated spring. The viscous drag of the fluid against the spindle deflection was measured by the spring deflection. Spring deflection was measured with a rotary transducer, which provides a torque Signal. The measurement range of the digital electronic viscometer was determined by the rotational speed of the spindle, the size and the shape of the spindle rotating, and the full-scale torque of the calibrated spring.

DETERMINATION OF DENSITY, SPECIFIC GRAVITY AND °API OF CRUDE OIL EMULSION SAMPLE
PROCEDURE

i. Determine and record the weight of the empty clean and dry pycnometer with stopper, W_1
ii. Fill the Pycnometer with oil sample, insert the stopper and wipe the Pycnometer exterior.
iii. Weigh the pycnometer with the oil sample sample and stopper, W_2
iv. Empty the pycnometer, wash it with clean water and fill with distilled water.
v. Weigh it as in the same way as in (iii), W_3

Note.
Weight of empty pycnometer with stopper = W_1 gm
Weight of pycnometer filled with oil = W_2 gm
Weight of pycnometer filled with distilled water = W_3gm

Calculations

Density of liquid is calculated from the Equations 1, 2 and 3:
Weight of oil sample = (W_2-W_1) gm
Weight of equal volume of water =$(W_3 -W_1)$ gm

$$\text{Density of oil, } \rho_o = \frac{(W_2 - W_1)}{(W_3 - W_1)} \text{gm/cc} \quad (1)$$

at room temperature

$$\text{Specific Gravity, S.G} = \frac{(W_2 - W_1)}{(W_3 - W_1)} \text{ at t °C} \quad (2)$$

$$\text{API Gravity} = \frac{141.5}{SG} - 131.5 \quad (3)$$

Convert this API gravity to room temperature (t ℃) to API gravity at 60°F from the chart, which comes to be API at 60°F.
Note: Ensure that no air is trapped in oil or water when filling the pycnometer

DETERMINATION OF BASIC SEDIMENT AND WATER (BS AND W)

Ten (10) ml of the top oil after treatment with the local demulsifier blends were collected from each of

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The thirty (24) bottles with a syringe just a little above the oil – water interface, poured into different Graduated centrifuge tubes and labeled accordingly. Five centrifuge tubes were placed at a time in a Centrifuge machine and whirled for 10 minutes.

The percentage value of sludge or sediment and water were recorded:

$$(\%v/v) = (\frac{v_1}{v_2} \times 100) \quad (4)$$

Where:

$= \text{volume of separated wated}$

$v_2 = \text{volume of original water}$

TEST PROCEDURE OF FORMULATED LOCAL DEMULSIFIER BLENDS AT ROOM TEMPERATURE

The performance of the different demulsifier blends were ascertained by contacting them with the crude Oil in a ratio 1:50 ml. 50 ml of the crude oil emulsion samples from lake oil Ikuru town.

Sample A was introduced into 5 bottles, and 50 ml of (sample B) was introduced into another 5 bottles.

Then, with the aid of a syringe, 1 ml of each of the four locally formulated demulsifier blends as well as the Patent commercial demulsifier used for 'control experiment' were then added to the ten (10) samples and the mixture were vigorously mixed to homogenize and left to stand at room temperature.

The process was monitored for four hundred and eighty (480) minutes at 30, 60 minutes interval.

II. RESULTS

Demulsfication of two samples of emulsified crude oil was achieved with the use of locally formulated demulsifier and commercial demulsifier. The analyses were conducted at temperatures of 20°C, 40°C, and 60 °C upon concentrations of 0 %, 1 %, 5 %, and 5 % respectively. The physical properties of samples of the emulsified crude oil, commercial demulsifier, and locally formulated demulsifier were experimentally analyzed so as to establish the quality of the products. The diagnostic tests conducted were the API, Specific gravity, Density, pH, and viscosity at a temperature of 60^0Fas shown in Table 3. The densities of both samples of crude oil were 64.05 g/cm meaning it is light crude oil. The API gravity and specific gravity of the crude oil (bottom) were 29.299 and 0.88; crude oils (top) were 33.03 and 0.86 respectively which also confirmed the crude oils were light. The pH of both crude oil and the locally formulated demulsifier was 7.0 meaning the two crude oil samples were neither acidic nor alkaline. The pH was analyzed because of its importance, that is, it is a key factor that determines the microbial growth and production of demulsifiers. It then means that the locally formulated demulsifier is free from the bacterial attack which is also in line with the findings of[4] which state that a pH of 7 or close to 7 is recommended in the majority of the cases for producing emulsifiers. The API gravity ad specific gravity of the commercial demulsifier and locally formulated demulsifier were 36.94 and 0.84; 41.06 and 0.82 respectively. The experimental results had shown that the commercial demulsifier is lighter than the locally formulated demulsifier. The temperature, mixing ratio, and solubility of the locally formulated demulsifier may have contributed to the high API gravity value as observed by other authors. And also, to the concentration of the hydrophilic end used in the formulation. The experimental results of the physical properties have shown that there was a slight difference between the commercial demulsifier and the locally formulated demulsifier.

Table 3: Physical Properties of Crude Oil Samples, Commercial and Locally Sourced Demulsifiers;

S/N	Physical Properties	Crude Oil (Bottom)	Crude Oil (Top)	Commercial Demulsifier	Local Demulsifier
1	Density	64.05g/cm	64.05g/cm	NIL	NIL
2	Specific Gravity	0.88	0.86	0.84	0.82
3	API Gravity	29.299	33.03	36.95	41.06
4	pH	7.0	7.0	6.8	7.0
5	Viscosity	870kg/m	870kg/m	NIL	NIL

Rate of Separation with respect to Concentration of Emulsified Crude Oil with Local Demulsifier and Compare with an Imported Sample

The rate of separation with respect to concentration is shown in Figures 1 to 6 and the analyses were conducted at temperatures of 20°C, 40°C, and 60 °C respectively. Upon 0 %, 1 %, 5 %, and 10 % concentration at 10 minutes rate of separation results obtained with the local demulsifier and commercial demulsifier on the emulsified crude oil (top) at a temperature of 20 ^{0}C were 4 ml, 3.2 ml, 3 ml, and 4 ml and 2 ml, 1.8 ml, 2.5 ml, and 2 ml whereas on emulsified crude oil (bottom) were 5.5 mi, 2.5 ml, 4 ml and 5 ml and 4.5 ml, 4 ml, 4.5 ml, and 5 ml respectively as shown in Figures 1 and 2. Upon the same concentration, at 30 minutes rate of separation, results obtained with the local demulsifier and commercial demulsifier on the emulsified crude oil (top) at a temperature of 40 ^{0}C were 5 mi, 3 ml, 3 ml, and 4 ml and 2 ml, 3.5 ml, 2.5 ml and 2.5 ml whereas on emulsified crude oil (bottom) were 4.7 ml, 2 ml, 3.5 ml and 4.5 ml and 4 ml, 4 ml, 4.5 ml and 5 ml respectively as shown in Figures 3 and 4. Upon the same concentration, at 30 minutes rate of separation, results obtained with the locally formulated demulsifier and commercial demulsifier on the emulsified crude oil (top) at a temperature of 60 ^{0}C were 2 ml, 3.2 ml, 3 ml, and 4.5ml and 3 ml, 3.5 ml, 2.5 ml, and 3 ml whereas on emulsified crude oil (bottom) were 3 ml, 3.5 ml, 2.5 ml and 3 ml and 2.5 ml, 3.5 ml, 4.5 ml, and 6 ml respectively as shown in Figures 5 and 6. Experimental results showed that appreciable rate separation was obtained with both the locally formulated demulsifier and commercial demulsifier meaning that the demulsifiers were effective at the investigated temperatures and time.

[4, 5]. However, a linear rate of separation was obtained at 30 minutes at 60℃ which implies that temperature and retention time has an influence on demulsification operation therefore attention must be paid to these factors in the choice of demulsifier to be used which also the finding of other researchers.

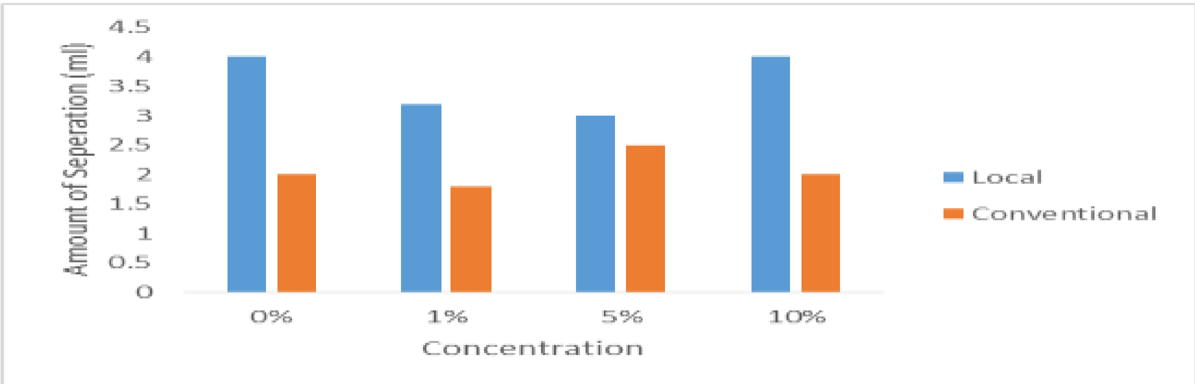

Figure 1: Results of Local and Conventional Demulsifier of 20 ^{o}C at 10 minutes Separation of Crude Oil at the Top

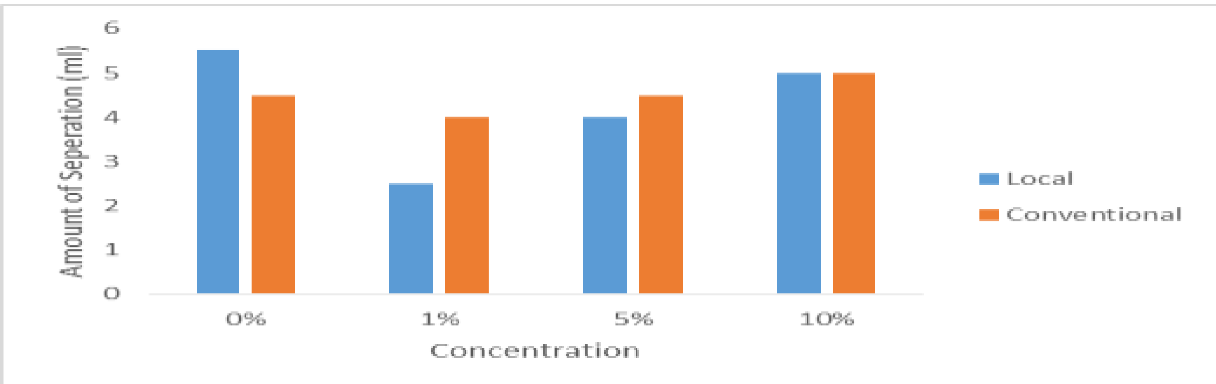

Figure 2: Results of Local and Conventional Demulsifier of 20 ^{o}C at 10 minutes Separation of Crude Oil at the Bottom

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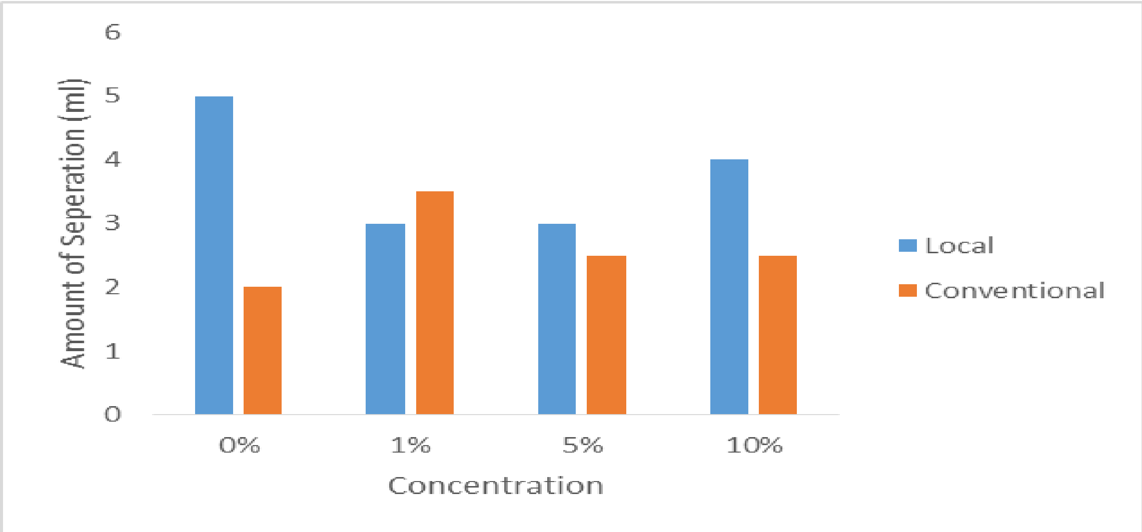

Figure 3: Results of Local and Conventional Demulsifier of 40 °C at 30 minutes Separation of Crude Oil at the Top

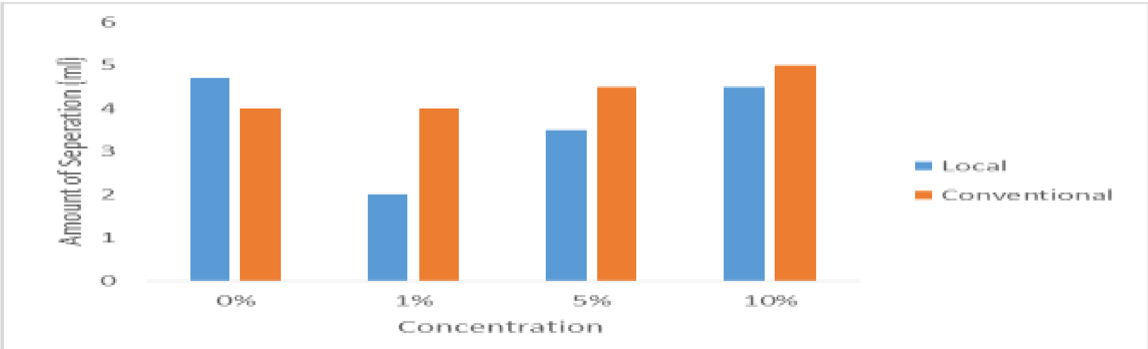

Figure 4: Results of Local and Conventional Demulsifier of 40 °C at 30 minutes Separation of Crude Oil at the Bottom

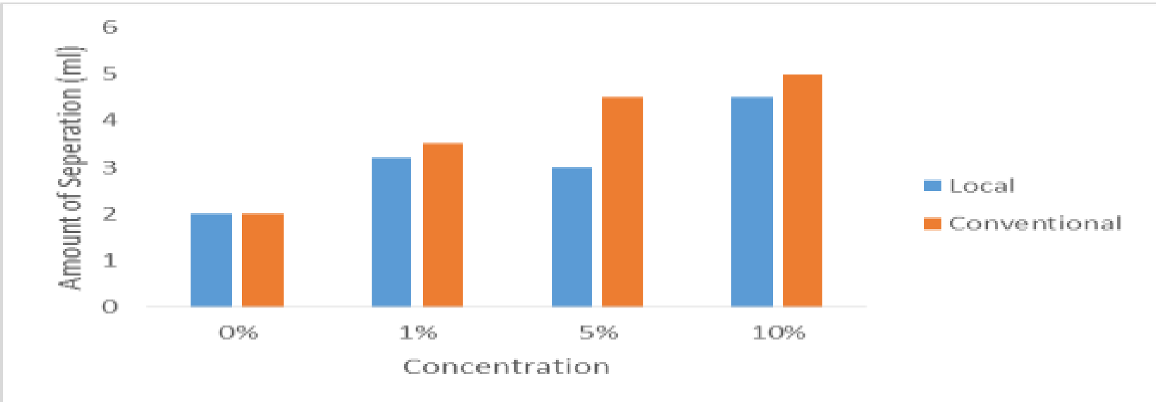

Figure 5: Results of Local and Conventional Demulsifier of 60 °C at 30 minutes Separation of Crude Oil at the Top

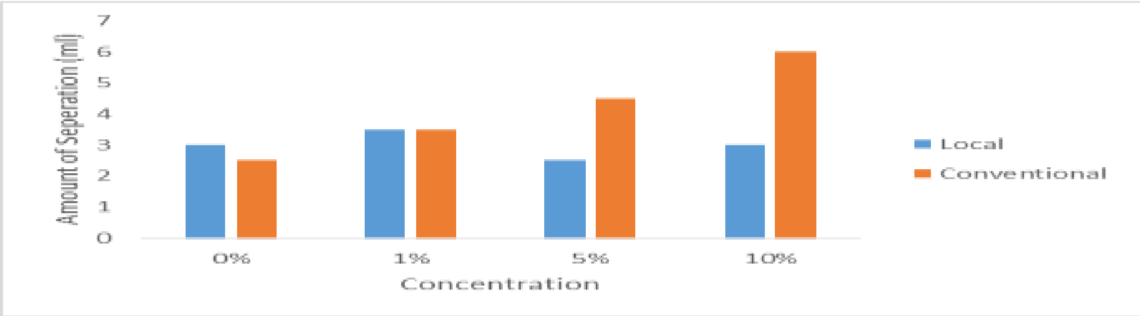

Figure 6: Results of Local and Conventional Demulsifier of 60 °C at 30 minutes Separation of Crude Oil at the Bottom

III. CONCLUSION

1. Experimental results have showed that local demulsifier can be highly effective with low separation time in separating oil from water in oil-water emulsions.
2. Imported and local demulsifiers spontaneously resolved the emulsion in the crude oil sample producing nearly 100% dry crude oil with trace elements.
3. Some local demulsifiers can perform better than some imported demulsifiers in resolving oil-water emulsions into their separate constituents.

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