

Stability Study of Some Selected Nigerian Crude Oil Emulsions and the Effectiveness of Locally Produced Demulsifier

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Abstract

Crude oil emulsion stability causes high viscosity of crude oil which leads to problems in pipeline transportation and processing. In this research, four samples of crude oil emulsions were collected from Ughelli East Wellhead and labeled A, B, C and D. These samples were characterized to determine their specific gravity, ⁰API gravity, structural composition, density, temperature, amount of water, amount of gas and the gross crude oil production. The ⁰API at 60 °F showed that all the samples had values below 30 ⁰API which was an indication that the crude oil produced from these wells were heavy crude oil. The FTIR spectra showed bands at 3444.55 cm⁻¹ for Sample A, 3418.23 cm⁻¹ and 3175.94 cm⁻¹ for Sample B, 3444.61 cm⁻¹ for Sample C and 3444.49 cm⁻¹ for Sample D attributing to strong and broad O-H stretch, H-bonded and an indication of Asphaltenes presence; the alkanes signal, C-H stretch at 2961.14 cm⁻¹ for Sample A, 2926.00 cm⁻¹ for Sample B, 2924.15 cm⁻¹ for Sample C and 2922.88 cm⁻¹ for Sample D are indications of the presence of wax; C=O stretch at 1737.74 cm⁻¹ only present in sample C is an indication of the presence of resins. The well test details showed that sample A and B had smaller water percentage in the gross crude oil production than Sample C and D with high water percentage. Therefore crude oil produced from these four wells represented by samples A, B, C and D contains natural emulsion stabilizers and stable. However, the stability of Samples A and B are more than that of Samples C and D. The performance of locally produced demulsifier increased with increase in residence time of contacting it with the emulsion samples at their operating temperatures, while equal dosage of patent Separol N46 demulsifier showed no performance within the same residence time. However, treatments A_{WHT}, B_{RT}, C_{WHT} and D_{WHT} all at well head temperatures of 35.6 °C, 27 °C, 48.2 °C and 48.8 °C respectively showed that most water was expelled in 480 minutes.

Key words: Stability, Selected, Crude oil, Emulsion, Demulsifiers, Treatment

1. Introduction

Crude oil is a complex mixture of saturates (paraffin/waxes) aromatics, naphthalenes, asphaltenes and resins (Lee, 2008). In petroleum industry, the viscosity of crude oil is the crucial part to investigate. The viscosity of crude oil in industry is about flow properties of emulsion which involve two phase flows between water and crude oil. Emulsion is a system with dispersion of a liquid phase into another. Water is normally present in crude oil reservoirs or is injected to stimulate oil production and pipelining, and they include those of water-in-oil (W/O), oil-in-water (O/W) and more complex oil-in-water-in-oil (O/W/O) emulsions. Hence, such emulsions are detrimental to oil production since oil viscosity raises increased corrosion issues and are difficult to break in desalting and dehydrating units before refining. An oil-in-water emulsion is a mixture of two immiscible liquids where oil phase is dispersed into the water continuous phase. Oil-in-water emulsions are very rare, deliberately produced to reduce the viscosity of highly viscous crude oil so that they can be transported easily through pipelines (Zaki, 1997). The oil-in-water emulsion reduces the viscosity of heavy crude oils and bitumens and may provide an alternative to the use of diluents or heat to reduce viscosity in pipelines (Langevin *et al.*, 2004). Stability is a consequence of the small droplet size of water and the presence of an interfacial film on the droplets in emulsion, which make stable dispersions. Emulsion stability is considered in three different processes which is creaming, aggregation and coalescence. There are two main factors that affect the stability of crude oil emulsion and they are viscosity and density. A crude oil emulsion can be stable, unstable and meso-stable (Lee, 1999). Demulsification is a process of breaking emulsions in order to separate water from oil, which is also one of the first steps in processing the crude oil after transportation from reservoir. The quality of crude oil is highly dependent on the residual contents of water and water-soluble contaminants which will be problematic for the water treatment part of the processes (Lee, 1999). Chemical demulsification is the most widely used method to treat crude oil-in-water (O/W) and water-in-crude oil (W/O) emulsions.

Therefore, in this research, four samples of crude oil emulsions shall be collected from Ughelli East Wellheads and labeled samples A, B, C and D. The physical characterization of the crude oil emulsion shall be carried out to determine their specific gravity, ⁰API gravity. Structural characterization shall also be carried out using Fourier Transform Infra-Red Spectroscopy (FTIR). Local demulsification shall be carried out using locally produced demulsifier from Jatropha seed oil, Cassava starch, Candle wax, Camphor and Liquid soap, and its effectiveness compared to that of patent commercial demulsifiers.

2. Statement of theory

Crude oil produced in Nigeria contains emulsions that are stable, unstable and meso-stable. These stabilities persist for hours, days, weeks and longer, and cause high viscosities of these crude oil which in turn lead to problem in their pipeline transportation, and high pressure drops in pipe flows of oils. Therefore the study of these emulsion stability limits understanding of interaction between different solubility classes, whether competitive or synergistic and aids in determining adequate demulsifiers that should be employed for their treatment.

3. Materials and methods

3.1 Materials

Materials used for this research are summarized in Tables 1 and 2.

Table 1: Materials for the stability study of crude oil emulsions, treatment and their uses

S/N	Name of Apparatus	Description/Model	Use
1	Fourier Transform Infra-Red (FTIR) Spectroscopy	Thermo Scientific NICOLET IS10	Determination of functional groups
2	Density Bottle	Surex, England	Determination of specific gravity
3	Hot plate	Gallenkamp, England	For heating
4	Multi mixer	Gallenkamp, England	For blending
5	Weighing balance	Scout Pro, Ohaus, London	For weighing
6	Measuring cylinders	Pyrex Technico, England	For measuring
7	Beakers	Pyrex, England	For weighing/blending
8	Syringe	Axojet syringe set, China	For suction
9	Centrifuge machine	Pyrex, England	For centrifuging
10	Graduated bottles	Pyrex, England	For mixing demulsifiers and emulsion
11	Thermometers	Pyrex, England	For measuring temperature
12	Plastic buckets	DANA plastic, Nigeria	For storage
13	Stirrer	Chemglass, New Jersey	For stirring
14	Water bath	Lindberg/Blue M Asheville, NC, USA	For indirect heating at constant temperature
15	Stop Watch	Quartz, China	For timing

Table 2: Materials for local demulsifier production and their importance

S/N	Materials	Code	Source	Location	Importance
1	Jatropha oil	Jo	Jatropha curcas seeds	Auchi	Non-edible green oil that acted as solvent for camphor and also increased the lipophilic properties of the crude oil
2	Camphor Powder	C	Conifer/Pinene tree (Turpene)	Warri	This formed the lipophilic end of the local demulsifier produced
3	Paraffin wax	Pw	Petroleum wax	Warri	This served as the bulking agent in the locally formulated produced
4	Starch	S	Cassava	Warri	This formed the hydrophilic end of the locally Produced demulsifier because of its strong affinity for water
5	Liquid soap	Ls	Saponification of fatty acids and alkaline	Warri	This served as the binder for the demulsifier produced from locally sourced materials to bind the lipophilic and the hydrophilic end
6	Distilled Water	Dw	Steam	Warri	This was used as solvent for the starch solution

3.2 Methods

3.2.1 Determination of specific gravity and ⁰API

The specific gravity and ⁰API were determined using IS: 1460-2000 method. An empty density bottle was weighed. The same density bottle was filled with water and weighed. The density bottle was again filled with the same volume of crude oil emulsion as that of the water and weighed. The specific gravity was calculated using equation (1).

$$\text{Specific gravity} = \frac{\text{Weight of Crude oil emulsion}}{\text{Weight of equal volume of water}} = \frac{W_3 - W_2}{W_1} \quad (1)$$

Where,

W_3 = weight of density bottle and crude oil emulsion

W_2 = weight of empty density bottle

W_1 = weight of equal volume of water

The ⁰API was then calculated from the specific gravity using equation (2)

$${}^0\text{API} = \frac{141.5}{\text{Specific gravity @ } 60^\circ\text{F}} - 131.5 \quad (2)$$

3.2.2 Well test details

Details of the well test carried out on the crude oil from the wellhead for each of the crude oil sample are shown in Table 3. They included test for density, temperature, amount of water, amount of gas, amount of oil and the gross crude oil production.

3.2.3 Structural characterization using Fourier Transform Infrared (FTIR) Spectroscopy

Infrared spectra were obtained for the samples of crude oil emulsion using a Thermo Scientific NICOLET IS10 FTIR spectroscopy, controlled by an Opus / IR software, which uses Fourier Transform to process data. Samples were analyzed as liquid films in KBr cells and dry KBr pellets.

3.2.4 Extraction of jatropha oil

The oil was extracted using standard mechanical method (hydraulic pressing) as described by A.O.A.C., 1990. 20 kg of Jatropha curcas seed was collected around Auchi in Edo State and dried for 20 days under sun light thus exposing the endocarp. The endocarps of the dried seeds were broken and the seeds collected and further dried in sun light for 7 more days and ground. 50 g of the ground jatropha seed was placed in a perforated medium called baft. The baft containing the sample was placed into an extraction pan with cover. The extraction pan with the baft were then placed into a vertical hydraulic press at a temperature of 60 °C. Pressure was slowly applied in the

range of 100 – 300 lbs/sq. for 80 minutes for the first baft oil extraction, 32 minutes for the second baft oil extraction and 25 minutes for subsequent baft oil extraction.
The percentage of oil recovered was calculated as follows:

$$\% \text{ of oil recovered} = \frac{\text{Weight of oil}}{\text{Weight of ground sample}} \times 100 \% \quad (3)$$

3.2.5 Production of liquid soap from locally sourced materials

125 g of Sodium Hydroxide (NaOH) was soaked in 150 g of distilled water for 24 hours in a bowl. Meanwhile, a solution of 192 g of Sodium Laureen Sulphate (SLS), 270 g Sodium Trioxocarbonate (iv) (Na_2CO_3) and 520 g of Sulphonic acid was prepared in another bowl. The solution of sodium hydroxide and distilled water was then added to the solution of sodium laureen sulphate, sodium trioxocarbonate (iv) and sulphonic acid. The mixture was gently stirred and more distilled water added to produce a homogeneous mixture of 10 liters with a pH of 9.0.

3.2.6 Production of local starch from cassava

Some quantity of dry cassava starch was purchased from a local market around Effurun in Warri. 262 g of the dry cassava starch was dissolved in 261 g of cold distilled water to form a solution. Meanwhile, some quantity of distilled water was made to boil on a gas burner at 100°C . 300.9 g of the boiled distilled water was added to the starch solution and gently stirred to form a paste- like solution.

3.2.7 Production of local demulsifier

25 g of camphor powder was measured and poured into a beaker containing 15 g of *Jatropha curcas* oil, placed on a heated hot plate at a regulated temperature of 40°C and gently stirred to completely dissolve the camphor. 10 g of paraffin wax was then added to the hot camphorated *jatropha* oil. Next, 30 g of prepared cassava starch was added to the mixture while stirring still continued. Finally, 20 g of prepared liquid soap was added to the entire mixture and stirred gently for 60 minutes on the heated hot plate to obtain a homogenous blend of local demulsifier.

3.2.8 Treatment of samples with produced local demulsifier at room temperature

The local demulsifier produced was contacted with the crude oil samples in the ratio 1:50 ml. 50 ml of the crude oil emulsion samples from Ughelli East stations W/10L (sample A) was introduced into 2 bottles, and 50 ml of W/10L (sample B) was introduced into another 2 bottles. Then, with the aid of syringe, 1 ml of the locally produced demulsifier was added to a sample each of samples A and B, as well as the patent Separol N46 demulsifier used as ‘control experiment’ was also added to a sample each of samples A and B, and the mixture were vigorously mixed to homogenize and left to stand at room temperature (27°C). The process was monitored for four hundred and eighty (480) minutes, first at thirty 30 minutes interval followed by 60 minutes intervals.

3.2.9 Treatment of samples with produced local demulsifier at wellhead temperature

The local demulsifier produced was contacted with the crude oil samples in the ratio 1:50 ml. 50 ml of the crude oil emulsion samples from Ughelli East stations W/10L (sample A), W/10L (sample B), W/8L (sample C) and W/1T (sample D) were each introduced into 2 bottles to make a total of 8 bottles. Then, with the aid of a syringe, 1 ml of the locally produced demulsifier as well as the patent Separol N46 demulsifier used as ‘control experiment’ (making a total of 2 demulsifiers) were then added to each of the 2 bottles containing the crude oil emulsion samples. The entire 8 bottles containing the mixture of the emulsion samples and the demulsifiers were vigorously mixed to homogenize and left to stand at regulated wellhead and water bath temperature of 35.6°C , for samples A and B respectively, wellhead temperature of 48.2°C and 48.8°C for samples C and D respectively in a water bath. The process was monitored for four hundred and eighty (480) minutes, first at thirty 30 minutes interval followed by 60 minutes intervals.

3.2.10 Determination of Basic sediment and water (BS and W)

Ten (10) ml of the crude oil emulsion samples A, B, C and D top oil after treatment were collected from each of the twelve (12) bottles with a syringe just a little above the oil – water interface, poured into different graduated centrifuge tubes and labeled as A_{RT} and A_{WHT} , B_{RT} and B_{WHT} , C_{WHT} , D_{WHT} , Separol and Blank accordingly. Five centrifuge tubes were placed at a time in the centrifuge machine and whirled for 10 minutes. The value of the basic sediment and water were read for each of the tubes and their percentage values recorded.

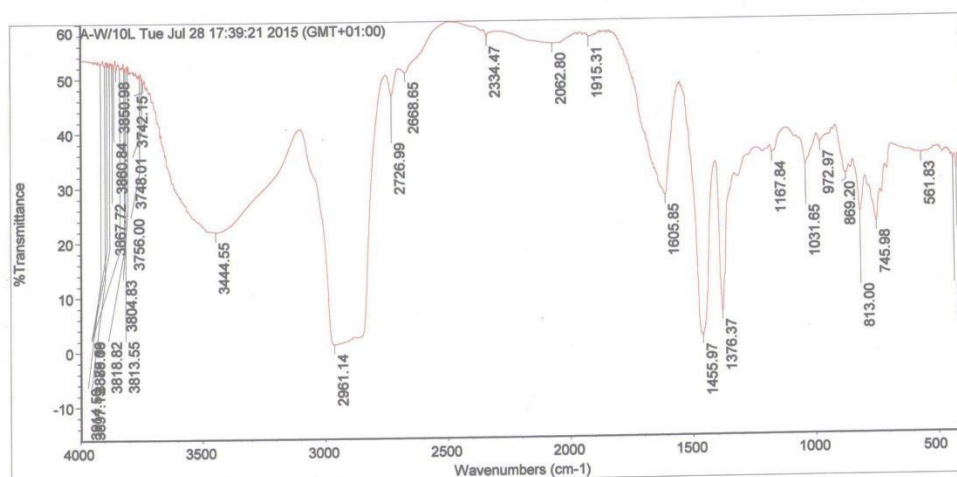
4. Results and Discussion

Results of the specific gravity and $^{\circ}$ API, and well test details are shown in Table 3. It showed that all the samples are heavy crude oil since their $^{\circ}$ API are all below 30° API. However, the water content in samples A and B is $\frac{1}{3}$ of the gross production and that of samples B and C is $\frac{2}{3}$ of the gross production.

Table 3: Samples test details of Ughelli East flow stations crude oil samples

Sample	Sample station	Gross bbls/d	Oil bbls/d	Water bbls/d	Gas/d mmscf	Temp $^{\circ}$ F	Specific gravity @ 60 $^{\circ}$ F	$^{\circ}$ API @ 60 $^{\circ}$ F
A	W/10L	329.26	225.89	104.37	0.12	96.10	0.995	10.71
B	W/10L	252.10	163.77	88.33	0.08	80.50	0.994	10.85
C	W/8L	1730.34	472.01	1237.33	0.34	118.70	0.915	23.14
D	W/1T	1849.60	520.29	1329.31	0.14	119.80	0.895	26.60

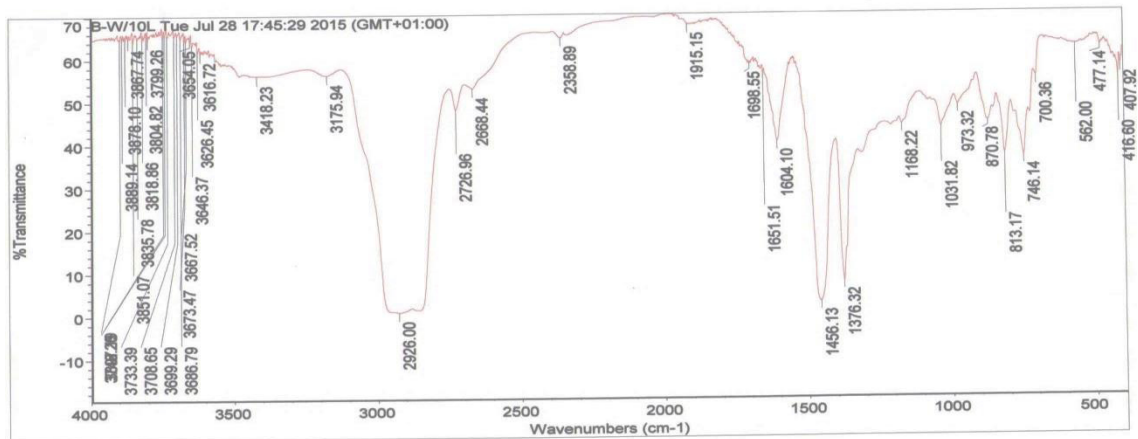
Also, Figures 1, 2, 3 and 4 shows the FTIR results of the samples. The spectra shows bands at 3444.55 cm^{-1} for sample A, 3418.23 cm^{-1} and 3175.94 cm^{-1} for sample B, 3444.61 cm^{-1} for sample C and 3444.49 cm^{-1} for sample D attributed to strong and broad O-H stretch, H-bonded and an indication of Asphaltenes presence; the alkanes signal, C-H stretch at 2961.14 cm^{-1} for samples A, 2926.00 cm^{-1} for sample B, 2924.15 cm^{-1} for sample C and 2922.88 cm^{-1} for sample D are indications of the presence of wax; C=O stretch at 1737.74 cm^{-1} only present in sample C is an indication of resins presence.



Tue Jul 28 17:41:10 2015 (GMT+01:00)
 FIND PEAKS:
 Spectrum: A-W/10L Tue Jul 28 17:39:21 2015 (GMT+01:00)
 Region: 4000.00 400.00
 Absolute threshold: 59.735
 Sensitivity: 89
 Peak list:

Position:	408.50	Intensity:	32.854
Position:	419.18	Intensity:	31.952
Position:	432.92	Intensity:	33.625
Position:	561.83	Intensity:	34.904
Position:	745.98	Intensity:	22.534
Position:	813.00	Intensity:	24.370
Position:	869.20	Intensity:	31.351
Position:	972.97	Intensity:	37.142

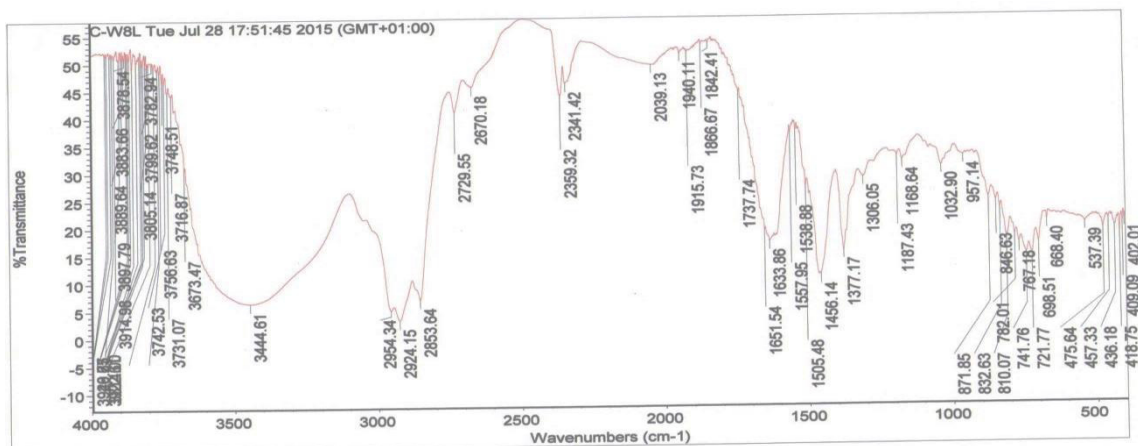
Figure 1: Fourier Transform Infrared (FTIR) spectrum for sample A



Tue Jul 28 17:46:59 2015 (GMT+01:00)
 FIND PEAKS:
 Spectrum: B-W/10L Tue Jul 28 17:45:29 2015 (GMT+01:00)
 Region: 4000.00 400.00
 Absolute threshold: 68.975
 Sensitivity: 68
 Peak list:

Position:	Intensity:
407.92	57.562
416.60	55.146
477.14	62.070
562.00	62.176
700.36	54.958
746.14	36.429
813.17	37.040
870.78	44.631

Figure 2: Fourier Transform Infrared (FTIR) spectrum for sample B



Tue Jul 28 17:53:41 2015 (GMT+01:00)
 FIND PEAKS:
 Spectrum: C-W/8L Tue Jul 28 17:51:45 2015 (GMT+01:00)
 Region: 4000.00 400.00
 Absolute threshold: 55.297
 Sensitivity: 75
 Peak list:

Position:	Intensity:
402.01	20.307
409.09	19.588
418.75	17.992
436.18	19.101
457.33	20.474
475.64	18.733
537.39	19.755
668.40	20.191

Figure 3: Fourier Transform Infrared (FTIR) spectrum for sample C

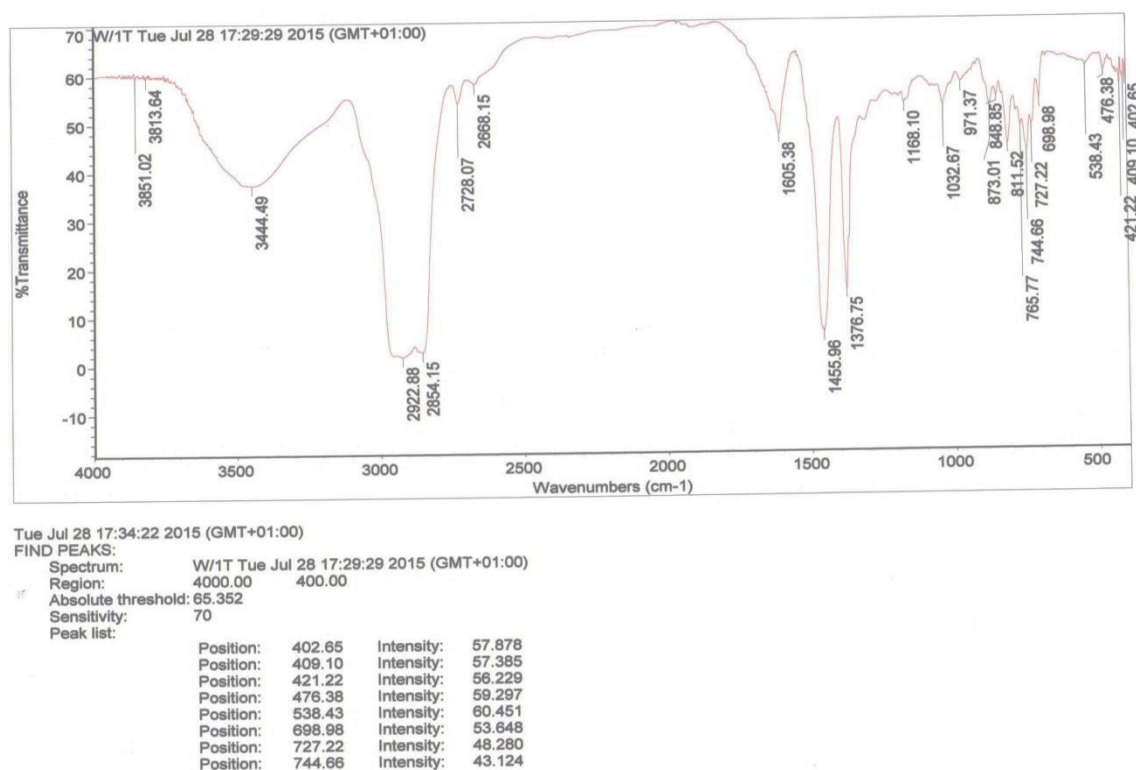


Figure 4: Fourier Transform Infrared (FTIR) spectrum for sample D

The oil content of *Jatropha curcas* was found to be 40.52 % which exceeded those of some conventional oil seed crops like cottons, olives, soyabeans, etc. (Pritchard, 1991). With this relative high percentage oil yield of 40.52 %, the processing of the oil for industrial purposes would be of economic importance. The performance of the locally produced demulsifier generally increased with separation time and temperature as shown in Tables 4 – 6 and Figures 5- 10. The local demulsifier showed better performance than patent separol within the residence time of 480 minutes considered for treatment. Its performance was also best at the well head temperature of the crude oil emulsion samples.

Table 4: Sample A– Ughelli East flow station W/10L well at room temperature (A_{RT}) and well head temperature (A_{WHT})

Separated water in ml. @ Room Temperature (27 ^o C) for sample A (A_{RT})				Separated water in ml. @ Well head Temperature (35.6 ^o C) for sample A (A_{WHT})		
Time (min)	Local	Separol	Blank	Local	Separol	Blank
30	0	0	0	0	0	0
60	0	0	0	0	0	0
120	0	0	0	0	0	0
180	0	0	0	0	0	0
240	3	0	0	0.5	0	0
300	5	0	0	5	0	0
360	6	0	0	8	0	0
420	8	0	0	8	0	0
480	10	0	0	10	0	0
	Clear	No Separation	No Separation	Partially clear	No separation	No separation

Table 5: Sample B– Ughelli East flow station W/10L well at room temperature (B_{RT}) and water bath temperature (B_{WBT})

Separated water in ml. @ Room Temperature ($27^{\circ}C$) (Well head temperature) for Sample B (B_{RT})				Separated water in ml. @ water bath Temperature ($35.6^{\circ}C$) for sample B (B_{WBT})		
Time (min)	Local	Separol	Blank	Local	Separol	Blank
30	0	0	0	0	0	0
60	0	0	0	0	0	0
120	0	0	0	0	0	0
180	0	0	0	0	0	0
240	1	0	0	1	0	0
300	5	0	0	3	0	0
360	5	0	0	3	0	0
420	8	0	0	4	0	0
480	10	0	0	5	0	0
	Opaque	No Separation	No Separation	Opaque	No separation	No separation

Table 6: Sample C and D– Ughelli East flow station W/8L and W/1T wells at well head temperature (C_{WHT} and D_{WHT} respectively)

Separated water in ml. @ Wellhead temperature ($48.2^{\circ}C$) for sample C (C_{WHT})				Separated water in ml. @ Wellhead temperature ($48.8^{\circ}C$) for sample D (D_{WHT})		
Time (min)	Local	Separol	Blank	Local	Separol	Blank
30	0	0	0	0	0	0
60	0	0	0	0	0	0
120	0	0	0	0	0	0
180	0	0	0	0	0	0
240	0	0	0	4	0	0
300	0.5	0	0	5	0	0
360	1	0	0	6	0	0
420	1	0	0	6	0	0
480	2	0	0	10	0	0
	Opaque	No Separation	No Separation	Opaque	No separation	No separation

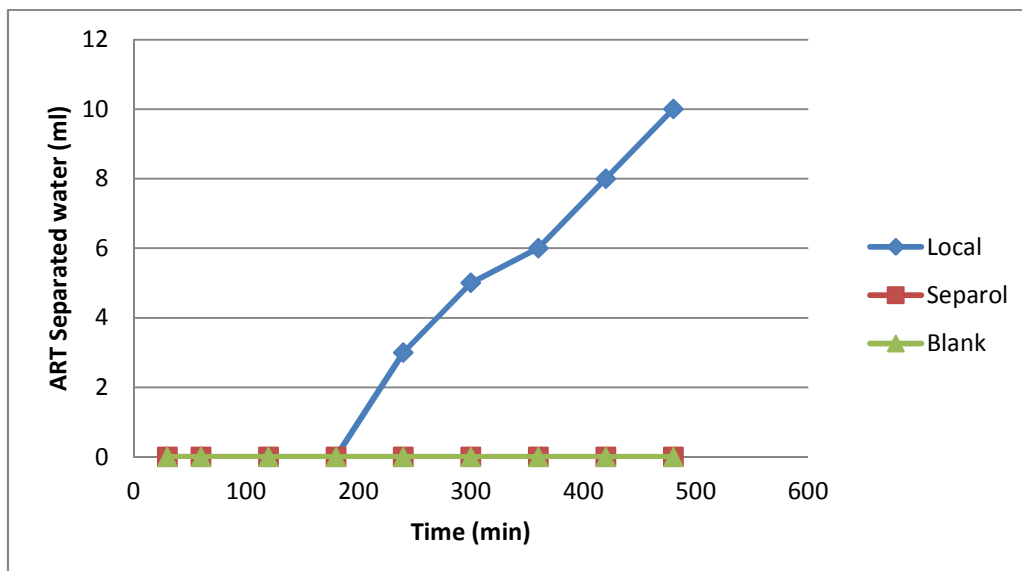


Figure 5: Performance of local and patent (Separol) demulsifiers on emulsion sample A at room temperature (A_{RT})

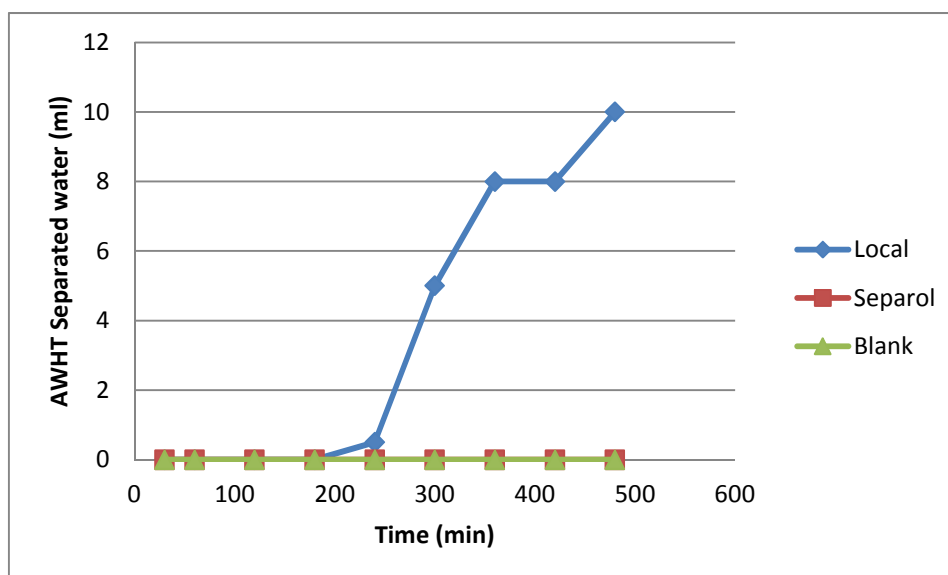


Figure 6: Performance of local and patent (Separol) demulsifiers on emulsion sample A at wellhead temperature (A_{WHT})

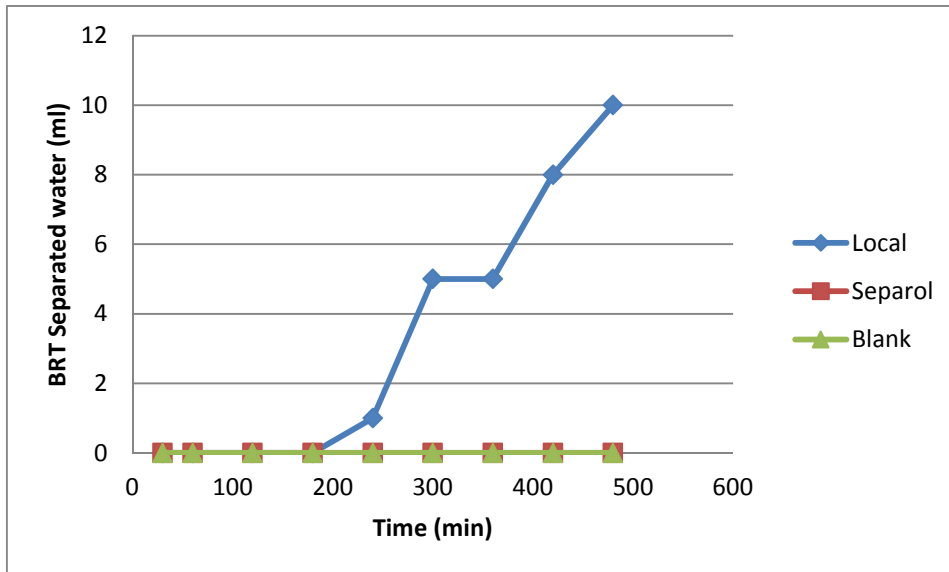


Figure 7: Performance of local and patent (Separol) demulsifiers on emulsion sample B at room temperature (B_{RT}) (Wellhead temperature)

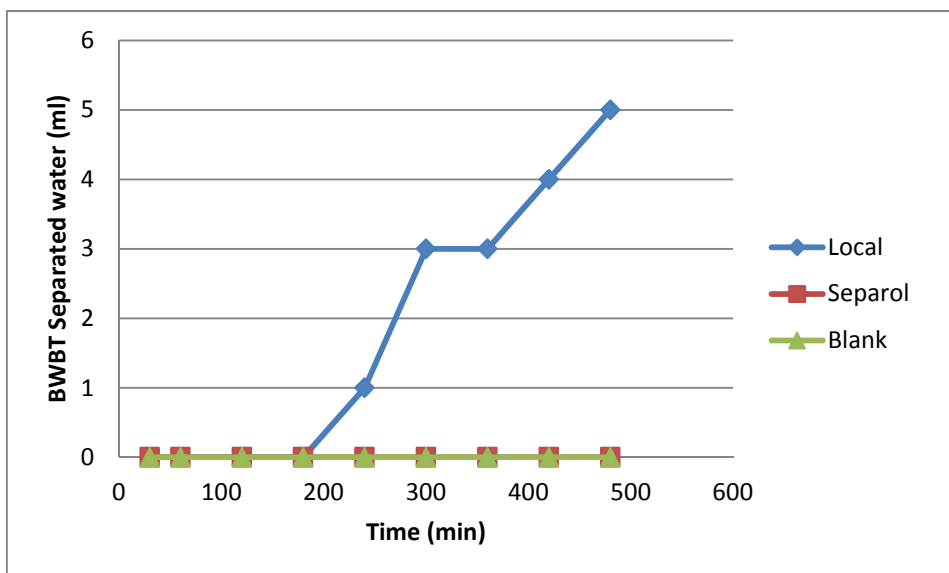


Figure 8: Performance of local and patent (Separol) demulsifiers on emulsion sample B at water bath temperature (B_{WBT})

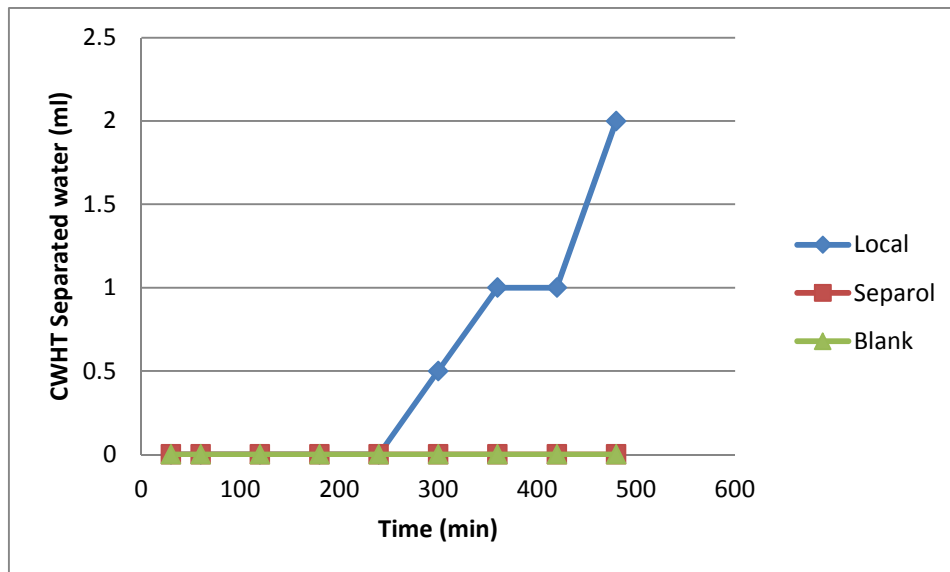


Figure 9: Performance of local and patent (Separol) demulsifiers on emulsion sample C at wellhead temperature (C_{WHT})

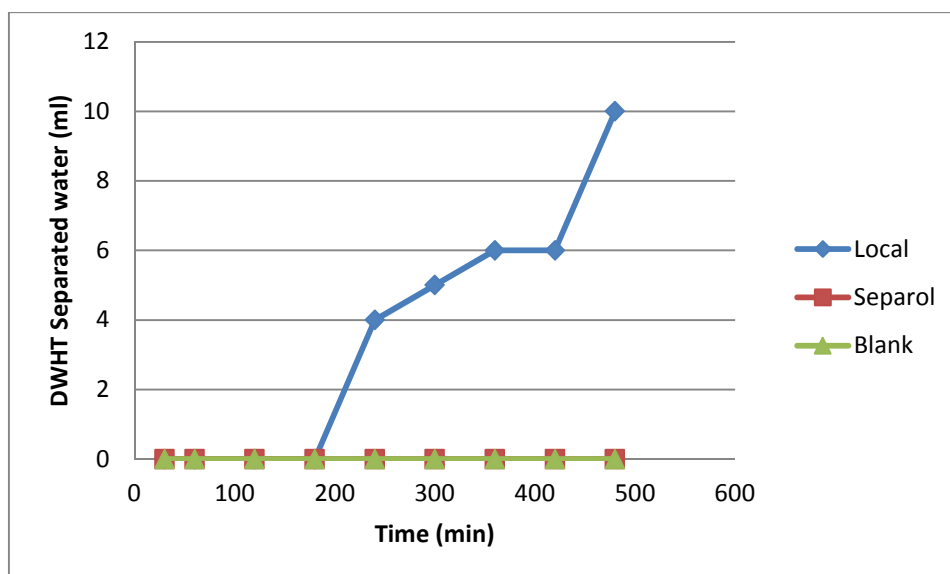


Figure 10: Performance of local and patent (Separol) demulsifiers on emulsion sample D at wellhead temperature (D_{WHT})

The effect of temperature on the performance rate of the locally produced demulsifier showed that treatments A_{WHT} , B_{RT} , C_{WHT} and D_{WHT} all at wellhead temperature expelled most water within the residence time of 480 minutes as shown in Figure 6, 7, 9 and 10.

5. Conclusion

1. Fourier Transform Infrared (FTIR) results showed the presence of natural emulsifying agents like asphaltenes and waxes in all the four crude oil emulsion samples studied except sample C that showed the presence of resins in addition to the asphaltenes and waxes. However, all four samples contained natural emulsion stabilizers.
2. All four emulsion samples showed $^{\circ}$ API gravity values below 30° API suggesting that they were all heavy and more stable crude oil samples. However, samples A and B showed more stability than samples C and D because of their lower $^{\circ}$ API gravity values of 10.71 and 10.85 respectively.

3. The well test details showed that samples A and B had smaller water percentage in the gross crude oil production than samples C and D with higher water percentage. These water percentages suggested that samples A and B had greater emulsion stability while samples C and D had lesser emulsion stability.
4. *Jatropha curcus* seed oil was a good source of solvent in the production of local demulsifier due to its high percentage yield and low moisture content.
5. Within the same operating conditions of demulsification of the crude oil emulsion samples, the patent (Separol) demulsifier did not separate any water, hence the efficiency of the locally produced demulsifier over the patent as shown in Tables 4 - 7 and Figures 5 - 10.

Table 7: Results of Basic Sediment and Water analysis samples A_{RT}, A_{WHT}, B_{RT}, B_{WBT}, C_{WHT} and D_{WHT} after treatment

Treatment	Basic sediment and water (%)	Top oil (%)
A _{RT}	0.8	99.2
A _{RT} S	10.0	90.0
A _{RT} B	10.0	90.0
A _{WHT}	0.5	99.5
A _{WHT} S	10.0	90.0
A _{WHT} B	10.0	90.0
B _{RT}	0.5	99.5
B _{RT} S	10.0	90.0
B _{RT} B	10.0	90.0
B _{WBT}	0.5	99.5
B _{WBT} S	10.0	90.0
B _{WBT} B	10.0	90.0
C _{WHT}	0.5	99.5
C _{WHT} S	10.0	90.0
C _{WHT} B	10.0	90.0
D _{WHT}	1.0	99.0
D _{WHT} S	10.0	90.0
D _{WHT} B	10.0	90.0

- A_{RT} : Sample A + Local demulsifier at room temperature
- A_{WHT} : Sample A + Local demulsifier at well head temperature
- B_{RT} : Sample B + Local demulsifier at room (well head) temperature
- B_{WHT} : Sample B + Local demulsifier at water bath temperature
- C_{WHT}: Sample C + Local demulsifier at well head temperature
- D_{WHT}: Sample D + Local demulsifier at well head temperature
- S: Samples + Separol at room, water bath and wellhead temperatures
- B: Blank samples at room, water bath and wellhead temperatures without demulsifiers

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