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EVALUATION OF THE PERFORMANCE OF DEMULSIFIERS IN THE TREATMENT OF CRUDE OIL EMULSIONS (A COMPARATIVE STUDY).

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ABSTRACT

Crude oil is often produced together with water (as free water and emulsion) from the reservoir. This water being undesirable must be removed using different methods and technologies to meet market specification. This research centers on the use of demulsifiers to aid the seperation of crude oil emulsion. A comparative analysis of the effect of five demulsifiers in treating crude oil emulsion from four different fields in the Niger Delta was done using the bottle test method. The best quality oil is oil with the lowest Basic Sediment & Water (BS&W) and best quality in terms of separated water volume. In the performance analysis, a combination of factors such as retention time, temperature effect and quality of separated water (sharpness of the oil-water interface and clarity of the separated water) were used to determine the best treatment. After the analysis, three demulsifiers (BCE 1052, IDD 2005 and TCE 1037) were able to separate the crude oil sample from okomo field and Imax field to $\leq$ 2% BS&W respectively. The emulsion sample from ADNB OML 123 field was demulsified but not within the $\leq$ 2% BS&W by any of the demulsifier. Based on the stated combined factors used in determining the best treatment, demulsifier IDD 2005 was adjudged the best suitable demulsifier, followed by BCE 1052 and TCE 1037. An optimum separation time of 10mins was obtained from OKOMO and IMAX field using IDD 2005, BCE 1052, and TCE 1037.

KEYWORDS: Demulsifies, bottle test, BS & W, emulsion, crude oil.

1. INTRODUCTION

Petroleum reservoirs usually contain water, oil and gas in its pore space. Of this three fluids, oil and gas (Hydrocarbon) is of interest to the petroleum industry while water which is usually produced alongside with the hydrocarbon is highly undesirable because of its effect on production facilities/equipment, cost of separation/deposal of the separated Water, and reduction of the quality of produced crude oil or natural gas (Pereira 2015; Wong 2015; Hanapi et al 2006).
Most times, this water is usually mixed with the crude oil forming a stable colloid Solution or emulsion and with natural gas as water vapor forming Hydrates. There are different types of crude oil emulsions. But for the

purpose of this study, concentration will be on water-in-oil emulsion as it is the most common during oil production (Abdulredha et al 2020). The Formation of Oil emulsion requires the presence of two liquid (oil and water), an emulsifying agent and enough agitation to ensure proper mixing (Raya et al 2020; John R. F. et al 2007).

The emulsification of Water in oil is normally difficult due to the immiscibility between the two liquid phases. However, shear mixing of the fluids during production and the existence of natural surfactants in the petroleum composition contribute to the formation of such emulsions (Raya et al (2020), Allenson et al (2011), Saad et al (2019)). Naturally occurring emulsifiers are concentrated in the higher boiling polar fraction of the crude oil and strengthens oil emulsion (Rondón et al 2008). These surfactants include asphaltenes, resins, and oil soluble organic acids (e.g. naphthenic carboxylic) and bases, which are the main constituents of the interfacial film surrounding the water droplets and the provides emulsion stability, hence retarding coalescence (Ekott and Akpabio 2011).

From the literature review, different Techniques and technologies such as Gas floatation, Ion-exchange, membrane filtration, electric separation etc. (Auther et al 2005; Abdulredha et al 2020) have been designed for the treatment of Oil emulsion, However, for effective separation and depending on the production design in place, these methods are often combined with specialized chemicals/demulsifiers and also optimizing some factors that affects emulsion stability such as reducing agitation, increasing temperature, and allowing sufficient time for separation (Al-Sabagh et al 2014).

DPR (2006, 2008) cited by Abdulkadir (2010) reported that the content of water (B.S&W) in oil ready for shipment is very important to oil producing companies. Many oil producing companies conduct measurements of the water content in crude oil automatically using the lease automatic custody transfer (LACT) unit, which passes oil to the pipelines only if the water content is below a preset maximum BS&W usually less than or equal to 1% (Larsen 2013).

Studies by salaam et al (2013), Singh, B. P. (1994) and Tambe, et al (1995) have emphasized the need for proper analysis of chemical demulsifies used in separating oil emulsion as the emulsion from different oil fields are not identical. Inappropriate demulsifiers can result in inefficient separation of the crude oil below market specification in addition to creating different problems such as formation of excess sludge layer, retaining oil droplets in the separated water resulting in increased cost for water treatment. This work investigates the performance characteristics of demulsifies in treating crude oil emulsion using five demulsifiers and crude oil samples from four field in Niger delta area of Nigeria. A careful consideration of this performance factors can guide production engineers in selecting the most appropriate demulsifiers for the treatment crude oil emulsion. However, factors such as concentration/dosage of the demulsifier was not studied due to the quantity of the available demulsifier and the effect of variation in temperature was not emphatically applied as the operator usually determines the test temperature based on the design separator operation.

2. MATERIALS AND METHOD

Crude samples from four different field were obtained and the effect of five demulsifiers on the crude oil emulsion from the fields was studied using the bottle test method. This method is the most common method used in oil/gas industry for reliable testing and ranking of demulsifiers based on its performance and also optimizing the factors that influences demulsification of the crude oil samples. However very rare cases have been reported where the bottle test result and field trial (designed based on the bottle test) result gave conflicting outcome (Johnson et al 2006). The apparatus used for the test include: water bath, stop watch, cylinders, centrifuge machines, beakers, syringe and needle, centrifuge tubes and micro pipette.

2.1. Sample Collection

The oil sample for the bottle test was collected from the well (wellhead sample) before been mixed with any chemical. This was to ensure a true representation of the crude from the well and that there is no contaminant whatsoever that could affect the results. However, the time interval from the time of collecting the crude oil emulsion to the time when the test was carried out was over 4 weeks due to procedural and internal challenges. The collected crude sample was properly cocked to prevent any foreign material from entering inside. The samples were taken to the laboratory and the total amount of water (BS & W) present in the emulsion sample was determined from the centrifuge test on the emulsion sample. Further experimentation was meticulously carried out. The parameters that were monitored and analyzed form the bottled test with the samples from the oil field using the five different demulsifiers includes: time of separation, separation temperature, rate of separation, cleanliness of separated water and sharpness of the interface between the water and crude oil. Table 1 shows the details of the crude sample before the bottle test experiment. The chemical demulsifiers used for the bottle test experiments are branded as IDD 2005, IDD 1903, BCE 1052, BCE 1045 and TCE 1837.

Table 1: Crude oil BS & W content

OIL FIELD/ OIL DATA	ADDAX OML 123 FIELD	OKOMO FIELD	OTAKIPOK marginal Field	IMAX
Well no.	ADNB-18	OKORO-14	13SS	
BS & W	26%	60%	5%	40%

2.2. Procedure for the Bottle Test

1. The crude sample was collected from the well head and poured into containers (care was taken to ensure that the sample was collected before been contaminated with chemicals).
2. Any excess water present in the crude was removed to the barest minimum before taken to the lab for testing.
3. The sample bottles were labelled with names of the demulsifiers and crude oil samples.
4. The container containing the crude oil emulsion sample was shaken vigorously and its content poured into centrifuge tubes for determining the BS & W
5. The crude oil emulsion sample was poured out to 50ml mark of the centrifuge tube and xylene was added to make up to 100ml mark. 20ppm of demulsifier was added to the tubes before centrifuging at 1500rpm for eight (8) minutes. The BS&W was obtained and recorded.

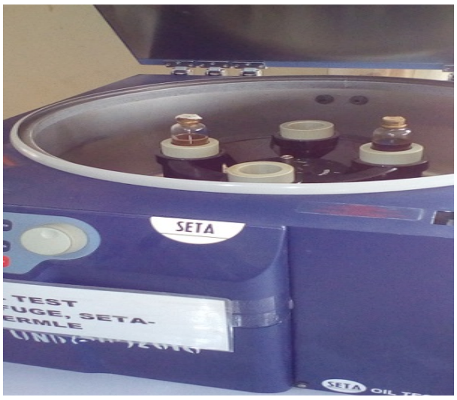

Figure 1. Centrigutation to determine the Initial BS & W

6. The water bath was set at a temperature of 52^0C and 100ml each of the different crude oil emulsion sample was poured into calibrated centrifuge bottle.
7. The centrifuge tubes were then inserted into the water bath for 1-2 hours to allow the crude oil to attain the temperaature of the water. A thermometer was used to confirm the temperature reading.
8. After confirming the temperature, A micro pipette was used to dispense the various range of demulsifier blends (5) at a dosage of 20ppm into the sample bottle. The mixture was shaken to ensure proper mixing and placed back into the water bath.
9. Observation and water drop out rate every 2, 5, 10, 20 and 30 and 40 minutes were recorded.
10. Percentage of water seperated = $\frac{Volume\ \ of\ \ seperated\ \ water}{Volume\ \ of\ \ sample\ \ (100ml)} x\ 100\%$
11. This process was be repeated for all the crude oil samples using the demulsifiers and readings were recorded.

2.4. Precaution taken during the laboratory experiment

1. It was ensured that error due to parallax was avoided when taken the readings on the centrifuge tube.
2. It was ensured that the right quantity (volume) of crude oil sample, demulsifier, and xylene was used when applied.
3. The centrifuge containing the crude oil and demulsifier was well agitated to ensure thorough mixing before readings.
4. The temperature was monitored to ensure consistency with the test temperature

3. RESULTS

The results of the analysis carried out using the bottle test method to evaluate the effect different demulsifiers in the treatment of crude oil emulsion are shown on Table 2 – Table 5 and Figure 2 – Figure 4. These presented results clearly shown the effect of separation/retention time on volume of separated water and also the quality of separated water in selecting the right demulsifier for crude oil treatment.

Table 2. Bottle test result and observation from OKOMO Field.

OKOMO FIELD-after centrifuge (BS&W 60%), color-brown, slug layer-44%						
		VOL% of water separated at 20PPM				
Time (min)	**Temperature (O*C)**	IDD 2005	IDD1903	BCE 1052	BCE 1045	TCE 1037
1	52	10	8	11	7	8
2	52	25	24	30	15	18
3	52	50	35	52	25	28
5	52	56	40	58	38	39
10	52	58	52	59	46	48
30	52	58	52	59	48	53
40	52	58	52	59	50	53
Quality of separated water						

	sharpness of Interface	Sharp	Sharp	Sharp	Slug layer	Sharp
	Clarity of separated water	Clear	Clear	clear	Not clear	Not clear

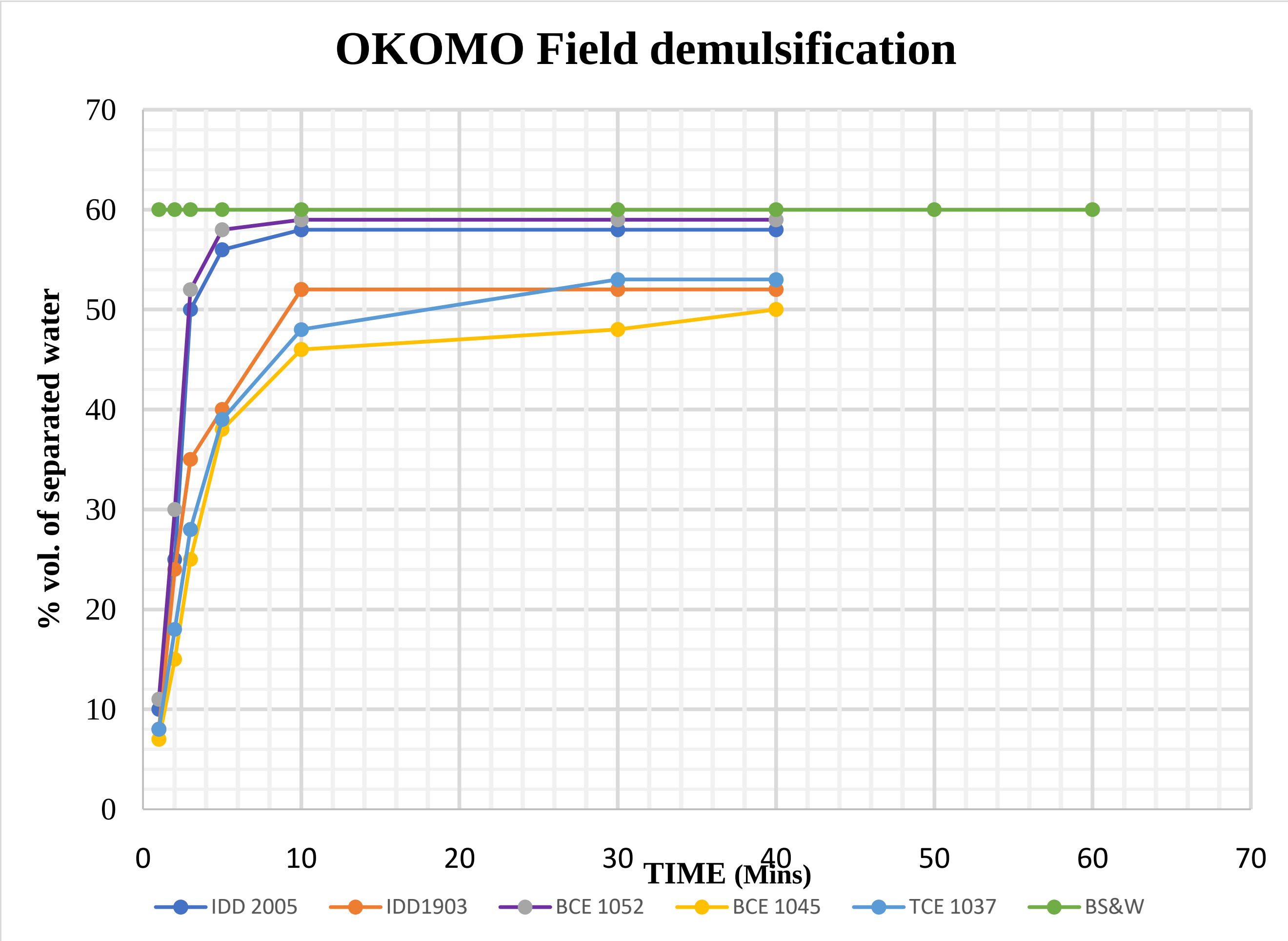

Figure 2. Okomo field demulsification

Table 3. Bottle test result and observation from IMAX field

IMAX FIELD-after centrifuge (BS&W 40%), color-dark grey, slug layer-39%						
		VOL% of water separated at 20PPM				
Time (min)	**Temperature (O*C)**	IDD 2005	IDD1903	BCE 1052	BCE 1045	TCE 1037
1	52	12.0	9.0	10.0	10.0	18.0
2	52	18.0	15.0	18.0	16.0	20.0
3	52	24.0	22.0	20.0	24.0	26.0
5	52	30.0	27.0	26.0	28.0	32.0
10	52	39.0	35.0	36.0	35.0	39.0
30	52	39.0	35.0	38.0	35.0	39.0
40	52	39.0	35.0	38.0	35.0	39.0
Quality of separated water						

	sharpness of Interface	Sharp	Slug layer	Sharp	Slug layer	Sharp
	Clarity of water	Clear	clear	Clear	not clear	Clear

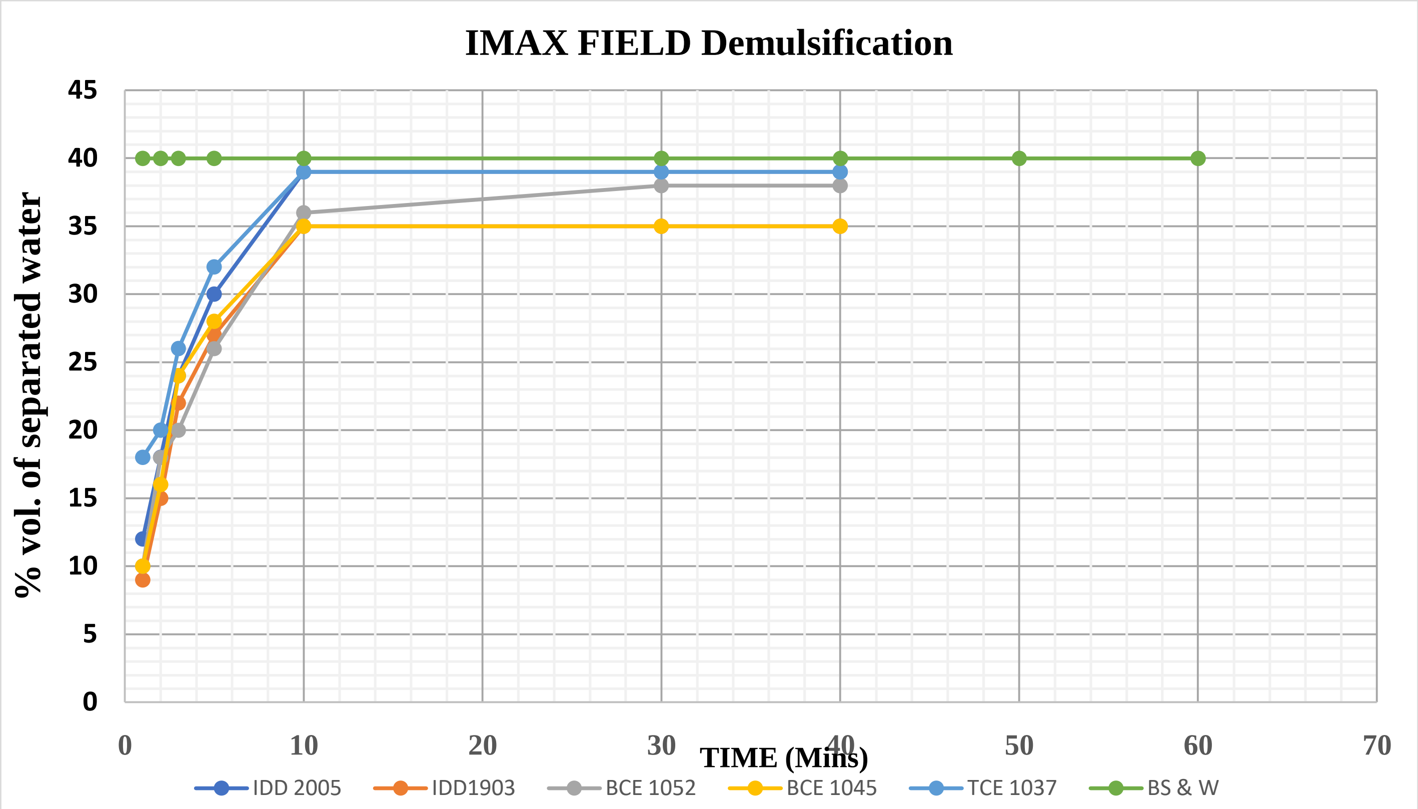

Figure 3. IMAX field demulsification

Table 4. Bottle test result and observation from ADNB OML 123 Field.

ADNB OML 123 FIELD-after centrifuge (BS&W 26%), color-dark brown, slug layer-24%						
Time (min)	**Temperature (O*C)**	**VOL% of water separated at 20PPM**				
		IDD 2005	IDD1903	BCE 1052	BCE 1045	TCE 1037
1	52	0.5	0.4	9.0	0.6	0.2
2	52	0.5	0.4	9.0	0.6	0.2
3	52	0.5	0.4	9.0	0.6	0.2
5	52	0.5	0.4	9.0	1.0	0.3
10	60	0.7	0.5	9.0	1.5	0.5
30	60	2.0	1.0	9.0	10.0	2.4
40	60	4.0	1.4	9.0	15.0	3.5
Quality of separated water						
	sharpness of Interface	Sharp	slug	Sharp	sharp	Slug
	Clarity of water	Clear	Not clear	clear	clear	Not Clear

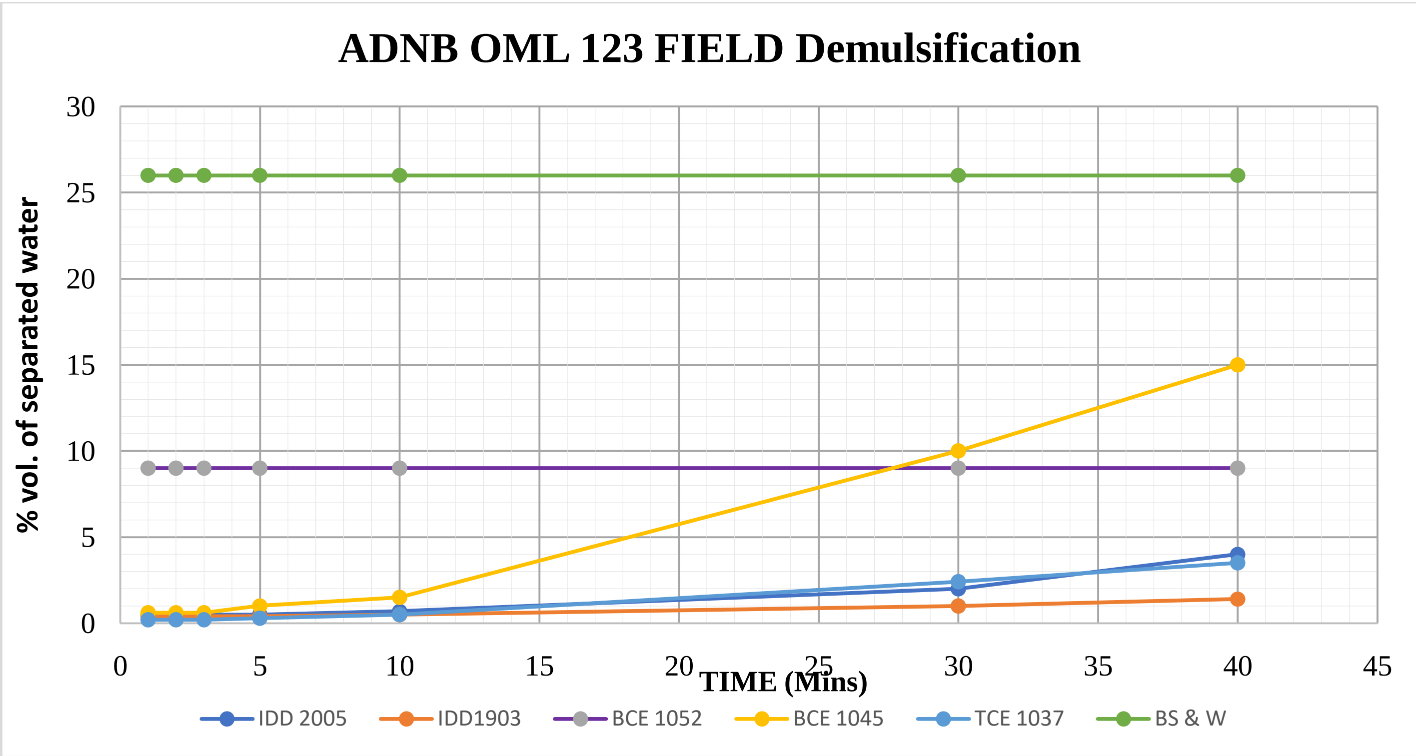

Figure 4. ADNB OML 123 field demulsification

Table 5. Table 4. Bottle test result and observation from Otakipok field

Otakipok Marginal field -after centrifuge (BS&W 0.5), color-black, slug layer-none						
Time (min)	**Temperature (O*C)**	**VOL% of water seperated at 20PPM**				
		IDD 2005	IDD1903	BCE 1052	BCE 1045	TCE 1037
1	52	0	0	0	0	0
2	52	0	0	0	0	0
3	52	0	0	0	0	0
5	52	0	0	0	0	0
10	52	0	0	0	0	0
30	52	0	0	0	0	0
Quality of separated water		NO Separation				
Clarity of separated water						

Figure 5. Clear water separated with sharp oil-water interface

4. DISCUSSION

4.1.Time of Separation

From the analysis performed, the maximum amount of time allowed for separation was 40 minutes. It can be observed from Table-2 (Okomo field) and Table-3 (Imax field) that after 10 minutes, the demulsifier IDD 2005 and BCE 1052 separated the water effectively and reduced the BS&W to a minimum of 2% and 1% respectively for Okomo field while demulsifier TCE 1037 and IDD 2005 reduced the BS&W to 1% for Imax field. Thus, the demusifiers were effective for reduction based on the time of separation. The other demulsifiers used for Okomo field and Imax field also separated significant level of water but not up-to or below 2% even after 40mins. Although increase in time allows for gravity settling, it is important to determine the optimum time that is economical. Decola and Popovich (2018) suggested that optimum retention time should be between 30mins to 60mins.

The idea of retention time having an effect on the volume of separated water is observed in Table 2 and table 3. The result from Table 2 (OKOMO field) and Table 3 (IMAX field) indicated that there was a substantial increase in the volume of the separated water as the retention time was increased from 1 to 10 minutes reducing the BS & W to less than 2% using IDD 2005, BCE 1052, and TCE 1037. This increase became insignificant after 10 minutes as no more separation was observed after 10mins. The result of ADNB OML 123 field (Table 4) shows a deviation from the value of the optimum retention time when compared to table-2 (Okomo field) and table-3 (IMAX field) while no separation occurred in table-5 (Otakipok Marginal field). The maximum volume of the separated water was 15% (BS & W--9%) using TCE 1045 for ADNB OML 123 field. Thus, for ADNB OML 123 field and Otakipok Marginal field, none of the demulsifiers were effective in reducing the water content as the BS&W value still remain high after separation for 40mins for ADNB OML 123 and none for Otakipok field. This could also be as result of aging of the crude oil which will be discussed in later session. The general observation from the results of table 2 to Table 4 shows that the higher the time of separation prior to the optimum separation time (10mins. for table 2 and 3), the higher the volume of separated water. However, after the optimum separation time, the volume of separated water increases insignificantly. For Okomo field, demulsifier BCE 1045 and TCE 1037 separated about 4% and 5% volume of water from the emulsion within 10min to 40mins compared to IDD 2005 and BCE 1052 (Okomo field and Imax field) and also TCE 1037(Imax field) with a retention time of 10mins. Hence demulsifiers with lower optimum time of separation are preferred. It is important to establish the optimum retention time for emulsion separation with the selected chemical demulsifiers as this will be taken into consideration when designing the separation time for separators to allow for sufficient demulsification before sending the crude oil to other treatment units. From the result of figure 2 and 3, one can confidently determine optimum retention time for any demulsifier in treating any given emulsion.

4.2.Effect of Temperature

Temperature used for separating crude oil emulsion is very important and affects the rate of demulsification. From table 4 (ADNB OML 123 field), after 5mins, water separation ceased, however, the process was repeated but the temperature increased after 5mins from 52^oC to 60^oC and separation was observed again.
This observation can be simply explained using density difference phenomena. When the crude emulsion is heated, it leads to viscosity reduction of crude oil emulsion with its resultant increase in the crude mobility, density difference supports gravity setting between the immiscible phases leading to coalescence and finally demulsification (Roshan et al 2018; Bi et al 2020). The application of heat also encourages proper mixing of

the emulsion with the demulsifier. However, depending on the separator design, care must be applied not to increase the temperature with the aim of increasing demulsification as high temperatures can lead to evaporation of light ends of the crude oil, reducing the API value, increasing the viscosity and can retard further demulsification as observed by Li et al (2018).

4.3. Effect of Aging

Tables 4 and 5 shows a pronounced deviation in the volume of separated water compared to table 2 and 3. This absence of separation (table 5) or reduction in volume of separated water (table 4) could be due to aging of the emulsion sample as the analysis was carried out after 4 weeks of collecting the sample. According to Maia Filho et al (2012) and Ferreira et al (2012), as Aging (Time duration between the date of sample collection and when the test was done) increases, the emulsion stability increases, reducing the efficiency of demulsifiers. Guillaume (2021) assert that aging hinders demulsifier efficiency by increasing the viscosity of the crude oil and also oxidation of aromatics and Sulphur containing compounds. Similar effect was observed by Maia Filho et al (2012) as no water was separated after 30 days during their study. However, table 2 (Okomo field) and 3 (Imax field) separated significant amount of water using the same demulsifiers implying that the effect of aging is dependent on the crude oil sample. However, for best result, it can be concluded that demulsifier performance analysis should be carried out promptly after collecting the crude oil samples.

4.4. Quality of Separated water

Excluding other factors, the quality of the separated water is important and determines the ranking of demulsifier performance in separating crude oil emulsion (Johnsen et al 2006; Ojinnaka et al 2015). The quality of the water can be qualitatively measured by considering the cleanliness of the separated water and the interface between the Oil and water.

4.5. Cleanliness of Separation of Water

The result from table 2 and figure 2 shows that BCE 1045 separated 50%. But the separated water was not clear as stains of crude oil was present in the water zone. This was also observed for IDD 1903 and TCE 1037 for ADNB OML 123 field and BCE 1045 for IMAX field. However, other demulsifiers such as BCE 1052 for Okomo field and BCE 1045 for ADNB OML 123 field separated out clear water from the crude oil emulsion. Clearly separated water reduces the cost of further treatment on the separated water before discharge as the water contains insignificant or no trace of oil.

4.6. Sharp water interface between the separated oil and water.

The sharper the oil/water interface, the easier the water can be physically removed from the oil with little or no treatment depending on the regulation in place. The result from the bottle test experiment shows that BCE 1045 and IDD 1903 for Imax field and IDD 1903 and TCE 1037 for ADNB OML 123 field showed slug layers between the separated oil and water interface. The slug layer contains a mixture of oil and water that need to be separated and treated before discharge. hence, this demulsifiers proved to be ineffective for breaking the crude oil emulsion in this fields. From the bottle test result, IDD 2005 and BCE 1052 for Okomo field, BCE 1045 and IDD 2005 from ADNB OML 123 field, IDD 2005 and BCE 1052 for Imax field, these chemical demulsifiers separated the emulsion without forming a slug layer interface.

5. CONCLUSION

1. It can be concluded that no one particular chemical demulsifier can treat all crude oil emulsion from different field due to difference in crude oil properties and content which varies from place to place. A particular chemical demulsifier may be effective in one or two field but should not be concluded as effective for other field until a test is carried out to confirm its effectiveness. Though not documented, However the author is conversant with field results where a particular demulsifier once effective for oil emulsion from a field proved to be ineffective on the same field later most likely due to injected chemicals, well workover/treatment techniques and recovery methods applied on the field that could have affected the properties of the oil and hindered the demulsifier.
2. The quality of the separated water is an important property to be considered in evaluating the performance of demulsifiers to reduce cost of water treatment after demulsification.
3. The effect of aging on demulsifier performance differs depending on the crude oil sample, however other studies agreed that best results for evaluating the efficiency of demulsifiers are gotten when aging is reduced to few days of obtaining the emulsion samples
4. From the analysis of result presented in this research, it can conservatively be concluded that IDD 2005 is adjusted best, followed by BCE 1052 and TCE 1037 for the chosen crude oil emulsion samples

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