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Hydrophobicity -Hydrophilicity Balance and Demulsification Potentials of Some Selected Chemicals

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ABSTRACT

Effective demulsifiers for the treatment of crude oil emulsions should consist of two ends – hydrophilic and hydrophobic. The choice and combination of the chemical components for optimum performance is dependent on the properties of the specific emulsion and cannot be generic. Failure to recognize this and the absence of a repository of information that matches the rheological property ranges of crude oil emulsions with the right balance of the chemicals has led to series of failed field and plant trials with attendant waste of time and money. This research focuses on the effect of the hydrophilic/hydrophobic balance on the performance of Epoxy resin alkoxylate and Alkoxylated alkyl phenol formaldehyde resin (hydrophobic) and Propylene oxide/ethylene oxide (PO/EO) polyol block copolymer and Amine-initiated polyol block copolymer (hydrophilic), blended in ratios of 20/80, 30/70, 40/60.50/50, 60/40, 70/30, 80/20. The epoxy resin alkoxylate/ (Propylene oxide/ethylene oxide) (PO/EO) polyol block copolymer were labelled A₁, A₂, A₃, A₄, A₅, A₆, A₇ respectively while Alkoxylated alkyl phenol formaldehyde resin/Amine-initiated polyol block copolymer were labelled B₁, B₂, B₃, B₄, B₅, B₆, B₇. The result shows that for crude A, a high hydrophobic/hydrophobic ratio is best while crude B-D show that a high hydrophilic/hydrophobic ration is the best.

1. INTRODUCTION

Crude oil is seldom produced alone because it is most times mixed with water in the cause of production. The water produced alongside the crude oil, together with the available emulsifying agents often cause the oil to form an emulsion. An emulsion is a heterogeneous liquid system consisting of two immiscible liquids with one of the

liquids (the water in this case) being dispersed as droplets while the other (the oil) is continuous (Jose and Tayfun, 2015; Monson, 1969). This water creates several problems which leads to the resultant increase in the unit cost of oil production or the operational expenditure (OPEX) arising from emulsion treatment, corrosion management and gross-fluid transportation.

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The presence and nature of emulsions define the technical approach adopted for their treatment. The separation of an emulsion can be achieved with any or a combination of methods such as: use of chemical demulsifiers (chemical treatment), heat application (thermal treatment), application of electrostatic fields (electrical treatment) or use of vessels with high residence time (mechanical treatment) (Jorge, et al., 2015). The mechanisms for emulsion destabilization by chemical demulsifiers which is widely adopted in most oil production and handling facilities involve flocculation, creaming, sedimentation and coalescence.

Over the years, chemical demulsification has assumed a very prominent place in crude oil treatment and there have been great strides in research and application of different demulsifier chemistries for the effective treatment of crude oil emulsions to get a market-ready crude oil (Zake, et al., 2000). These chemistries are the fundamental and basic building blocks for demulsifier formulation. These demulsifier chemistries include but not limited to phenol formaldehyde resin, alkoxyates, alkoxyated ethylene oxide/propylene oxide block polymers, alkoxyated alkyl phenols, alkoxyated amines, alkoxyated dicarbamates, alkoxyated diisocyanates, alkoxyated alkyl polyglycosides, alkoxyated polyethylene amines, alkoxyated polyethylene glycols, alkoxyated diglycidyl ethers, resin sulfonates and maleates, Phosphate esters of resins and glycols etc (Krittika, et al., 2014; Saad et al., 2020; Yau, et al., 2017; Abdel-Azim, 2010). The performance of a particular demulsifier formulation is a function of two fundamental factors: (i) the rheological properties of the crude oil

emulsion and (ii) compositional balancing of the demulsifier components or hydrophilic and hydrophobic composition (Martínez-Palou, 2015). Of interest in the application of demulsifiers is that a demulsifier chemistry could respond differently to different crude oil emulsions (field specific) because of the difference in the rheological properties of the crude oil emulsions and the emulsifying agents (Akpabio, and Ekott, 2013; Abdurahman, et al., 2007a; Abdurahman, et al., 2007b; Mosayebi, and Abedini, 2013). Therefore, a generic formulation of demulsifier in terms of components and the amount of each component will not be effective for all sources and types of crude oil emulsions. As a result of this challenge, oilfield chemicals application engineers are required to carry out in-situ bottle test and field trials to evaluate the suitability of the different demulsifier samples available so as to determine the one that is best fit for purpose for a particular field. Plant trials could be very expensive and tasking since it could run continuously for a period of twenty-four (24) to seventy-two (72) hours. Unfortunately, most oil producing companies will request that chemical vendors try their products on a NO-CURE NO-PAY basis reducing the risk on their part and transferring all liabilities of the trial to the vendor. This has been a cause of concern in the oilfield chemicals supply chain because, even after a seemingly successful bottle test, a subsequent plant trial may fail to meet the desired key performance index (KPI) because of several other factors with the vendor bearing all cost. Therefore, there is a need to develop a robust repository that contains information on emulsion stability index (based on emulsion causing properties or

rheological properties), composition of demulsifier concentrates (types and amounts) and established performance for the different classes so as to avoid guesses. This will help vendors to know what to apply in different fields and also help oil producing companies to be a bit specific in their request to vendors. This body of information is almost non-existent and needs to be developed. In this research work therefore, Epoxy resin alkoxylate (hydrophobic), Propylene oxide/ethylene oxide (PO/EO) polyol block copolymer (hydrophilic), Alkoxylated alkyl phenol formaldehyde resin (hydrophobic) and Amine-initiated polyol block copolymer (hydrophilic) were used.

2. MATERIALS AND METHODOLOGY

2.1. Collection of crude oil emulsion samples

The water-in-oil emulsions used in this study were obtained from four different crude oil wells located in different parts of Delta State, Nigeria. The samples were untreated and stable. The emulsions were collected with the aid of sampling cans. The process involved flushing of sampling lines to ensure that the samples were completely free from contaminants. Then, the sample cans were rinsed with the fresh emulsion samples two times to ensure that the cans are contaminants free before the samples were collected.

2.2. Formulation of treatment chemicals (Demulsifiers)

The treatment chemicals used were formulated by varying the hydrophobic (Epoxy resin alkoxylate and Alkoxylated alkyl phenol formaldehyde resin) and

hydrophilic (Propylene oxide/ethylene oxide (PO/EO) polyol block copolymer and Amine-initiated polyol block copolymer) compounds in a ratio of 20/80, 30/70, 40/60.50/50, 60/40, 70/30, 80/20. The epoxy resin alkoxylate/ Propylene oxide/ethylene oxide (PO/EO) polyol block copolymer were labelled A₁, A₂, A₃, A₄, A₅, A₆, A₇ respectively while Alkoxylated alkyl phenol formaldehyde resin/Amine-initiated polyol block copolymer were labelled B₁, B₂, B₃, B₄, B₅, B₆, B₇.

2.3. Bottle test screening

The demulsifiers formulated were screened using the bottle test method. The bottle test is an empirical test in which varying amounts of potential demulsifiers (formulated by varying the hydrophilic and hydrophobic compositional ratio) are added in specially graduated bottles (prescription bottles as shown in Figure 1) containing the sample of the emulsion to be treated. For each crude oil emulsion sample, 100mls each was measured into three (3) prescription bottles. Dosages of 20ppm, 30ppm and 40ppm of A₁ demulsifier formulation were applied with the aid of a micro pipette into the prescription bottles containing the crude oil emulsions and shaken vigorously for at least 5 minutes while degassing intermittently, and then allowed to stand for 40 minutes with an interval check of 10 minutes to take readings of the amount of water separated, unresolved emulsion and dry oil as the emulsion was being broken. After 30 minutes, it was shaken for another 1 minutes and allowed to stand for 10 minutes and the final reading taken. This step was repeated for A₂, A₃, A₄, A₅, A₆, A₇, B₁, B₂, B₃, B₄, B₅, B₆, and B₇.



Figure 1: Prescription bottles used for the test

2.4. Determination of Basic Sediment and Water (BS&W)

5 ml of the emulsion was taken out in a syringe and transferred into a 10 ml centrifuge tube. 5 ml of xylene (solvent) was then used to make up the volume (50/50). The tubes were shaken by hand for a proper mixture of the emulsion and then put in the centrifuge machine. The tubes were centrifuged for 5 minutes at 5,000 rpm (revolution per minutes) and the level of water was recorded. Same procedure was also followed to determine the BS&W of the treated oil with the top oil in focus (ASTM D4007-11).

2.5. Specific Gravity and API Gravity

A hydrometer was used in determining the specific gravity of the crude oil emulsion sample by measuring a volume of 200ml in a graduated cylinder and carefully dropping the hydrometer in it until it becomes stable. The relative density was then recorded (ASTM D4007-12).

2.6. Determination of Crude Oil viscosity

The standard test method for viscosity was used for this study. A rheometer with model number DV3TLVTJO was used to determine the viscosity of the crude oil emulsion sample. A beaker was put under the spindle and filled with crude oil emulsion until it touched the marked area

on the spindle. The spindle was set to rotate until the readings on display became constant and this was recorded (ASTM E3116-18).

2.7. Determination of interfacial tension of Crude Oil

Standard test method for interfacial tension of oil against water by the ring method was used for this study. A tensiometer with model number 1-800-458-2558 was used to determine the interfacial tension of the crude oil emulsion. A clean dish containing only water was set on the sample table. The sample table was raised until the ring became immersed into the water. The emulsion was poured on the surface of the water to a depth that prevented the ring from breaking the film at the surface. The dish was adjusted until the ring was at the interface and the lever arm was in neutral position. The torsion of the wire was increased and the dish lowered, keeping the index of the lever arm at zero. The interfacial tension of the emulsion was taken when the film at the interface was broken (ASTM D971-20).

2.8. Determination of Asphaltene content

A solvent extractor was used to determine the asphaltene content in the crude oil emulsion by measuring 20ml of crude oil into a glass flask along with 800ml of n-heptane, sealed with a stopper and mixed

thoroughly. It was allowed to equilibrate for two days and weighed. 100ml of the oil/precipitant mixture was poured into a filter paper clamped to a funnel cup and sealed with aluminium foil to reduce evaporation during filtration. A vacuum pump with rpm 1425 (manufactured by Edwards for gallenkamp) was connected to the side arm of the filtration flask to begin filtration. As the mixture passed through the filter paper, more oil/precipitant mixture

was added to the filter cup. After filtration, the filter paper, and the filtered asphaltenes were dried for five (5) days and weighed until the weight change was less than 0.0001g over a 12- hour period (ASTM D2007-80).

3. RESULT AND DISCUSSION

The initial properties of the four water-in-oil emulsion samples used for this study as can be seen in Table1.

Table 1: Properties of crude oil emulsions used for the analysis

Properties	Experimental Measurement Method	Sample A	Sample B	Sample C	Sample D
Viscosity (cP)	ASTM E3116 -18	396.9	26.1	17.21	7.8
Water content (%w/w)	ASTM D4007-11	40	30	60	80
Specific Gravity	ASTM D1298-12	0.98	0.94	0.92	0.91
API Gravity ($^{\circ}$ API)	ASTM D1298-12	13.35	19.4	22.7	24.4
Interfacial tension (dynes/cm ³)	ASTM E3116 -18	16	20	21	24
Asphaltene content (g/100ml)	ASTM D2007-80	0.57	0.43	0.26	0.21

Figures 1a-c, show the results obtained when different ratios of propylene-ethylene oxide were applied on crude oil emulsion sample A.

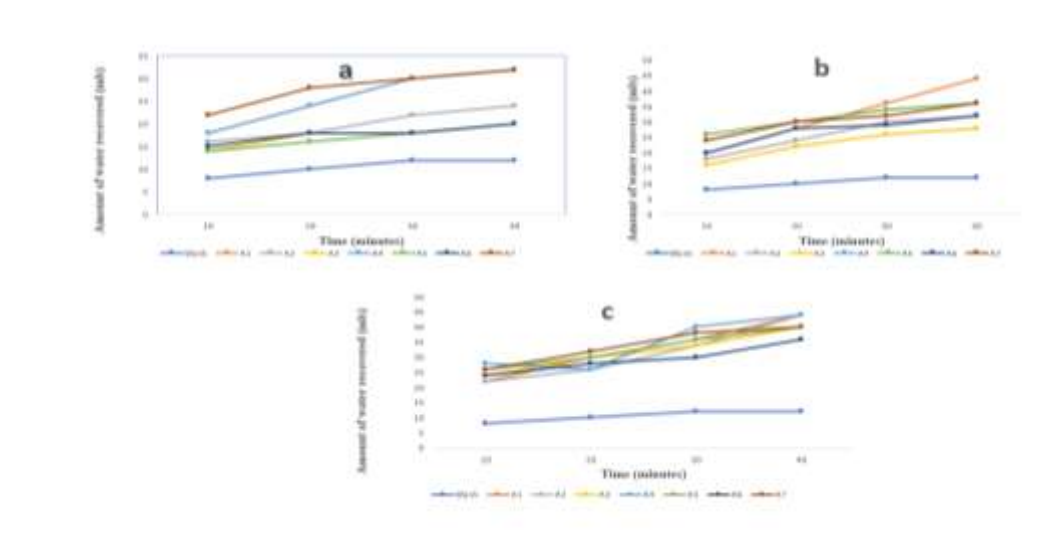


Figure 1(a-c): The effect of hydrophobic/hydrophilic balance on performance of demulsifiers A₁ to A₇ on crude A at (a) 20ppm, (b) 30ppm and (c) 40ppm

Figures 1a - c shows the results of the effect of the hydrophobic/hydrophilic balance (HLB) on the performance of demulsifier A₁ to A₇ when applied on crude A with rheological properties highlighted in Table 1 and at dosages of 20ppm, 30ppm and 40ppm with respect to amount of water recovered within specific separation times. The hydrophilic part is able to locate itself to the water molecule and the hydrophobic to the oil molecules present at the interface. From the results obtained, it shows that for crude A which is the crude oil emulsion sample with the highest stability (lowest interfacial tension), demulsifiers will require either low hydrophobic/hydrophilic ratio, the reverse or equal volumes and relatively low dosages in breaking the emulsions. For Figures 1 a - c, they were observed that at 20 ppm for every 10 minutes interval (10 - 40 minutes), A₁, A₄ and A₇ were more effective in breaking the emulsion represented by crude A, recovering 18ml, 24ml, 30ml and 32ml for A₁ and same for A₄ while 22ml, 28ml, 30ml and 32ml for A₇. At other dose rates (30ppm and 40ppm), demulsifier A₁ still showed relatively better performance giving 44ml after 30 and 40 minutes. This suggested that the formulation of demulsifiers could possibly swing between being a water dropper (high hydrophilic/low hydrophobic) or being an oil treater (high hydrophobic /low hydrophilic) but equal volumes will be recommended as depicted by A₇ (a good mixture of a water dropper and oil treater). These positions are premised on existing researches which showed that as hydrophilicity increases, performance increases and as hydrophobicity increases, performance will decrease. Sofiah, et al.(2020), describes demulsifiers as hydrophilic surfactants

while (Jorge et al. 2015 and Lixin 2021), reported that using a demulsifier with lower volume of hydrophilic base chemical will not effectively break the emulsion as this would leave water in the crude because the hydrophilic ends attach itself to the water medium and if the volume of hydrophilic base chemical is not sufficient, it cannot attract all the water molecules present in the emulsion and as a result reduces the market value, interferes with refining operations leading to more energy consumption in the breaking down of the crude oil and can induce corrosion in pipelines and vessels. Although Edith et al. (2022) suggested that hydrophilic demulsifiers are best suitable for oil in water emulsions while hydrophobic demulsifiers are better with water in oil demulsifiers. The result from this research differs from that position. It shows that since demulsifying chemicals can be blended to have both the hydrophilic and hydrophobic ends, a hydrophobic /hydrophilic ratio of 20/80 for crude with rheological properties within the bracket of what was used here will be adequate. Another look at the result showed that equal amount of the hydrophobic and hydrophobic based chemicals will also make a good blend.

Figures 2 a - c show the results of the effect of hydrophobic/hydrophilic balance on crude B with rheological properties highlighted in Table 1 and with different dose rates with respect to the separation time. The performance of the formulations on crude B (which is less stable and less difficult to separate compared to crude A) showed that A₁ and A₂ did better at 20ppm than the others. This suggested that at low dose rate, for emulsion within the properties

range of crude B, formulation with low hydrophobic/hydrophilic ratio did better.

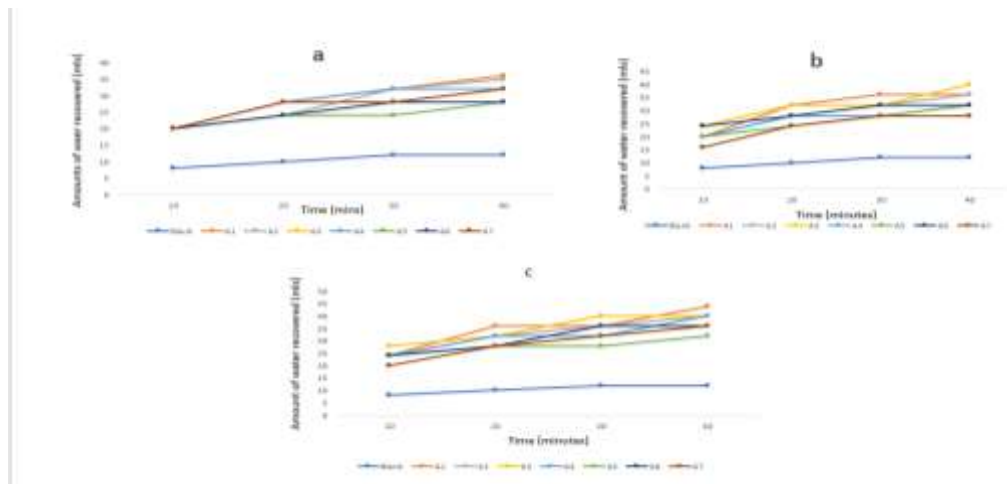


Figure 2(a-c): The effect of hydrophobic/hydrophilic balance on performance of Demulsifiers A₁ to A₇ on crude B at (a) 20ppm, (b) 30ppm and (c) 40ppm

This is in support of the argument advanced in the preceding section. For other dose rates (30ppm and 40ppm), it was observed that as the hydrophobic component increased the performances experienced a corresponding decrease. Kehinde et al. (2021), had investigated the performance of demulsifiers from different sources and concluded that the hydrophobicity and hydrophilicity characteristics impacted the performance of the different demulsifiers. In another research, Gandomkar, et al. (2020) reported that the nanoparticle based demulsifier with higher hydrophobicity characteristics enhanced water separation than regular deulsifiers with lower hydrophobic value because the nanoparticles had more chance to pass through the oil-water interphase films and reach the water phase and increase the density of water phase, thus, quickening the interfacial film rupture and oil water separation. However, this research shows

that considerable levy of hydrophobic characteristics will be required for emulsions that are tight and with high interfacial tensions, a higher hydrophilic characteristics value will be required (Rose et al., 2018). This is so since the emulsions are water – in - oil and will initially separate into three phases on treatment (oil, emulsion and water). The emulsion phase is laden with high volume of water and will require a good amount of hydrophilic chemical to attach itself with the water and enhance water molecules coalescence. In this way, the emulsion can be better treated producing dry top oil and water.

Figures 3 a - c show the results of the effect of hydrophobicity/hydrophilicity balance on crude C with rheological properties highlighted in Table 1 for different dose rates with respect to the separation time.

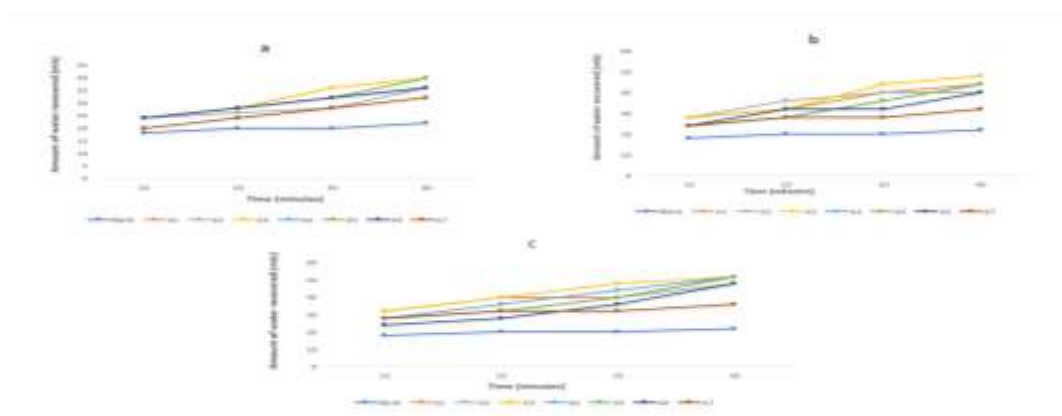


Figure 3 (a-c): The effect of hydrophobic/hydrophilic balance on performance of Demulsifiers A₁ to A₇ on crude C, at (a) 20ppm, (b) 30ppm and (c) 40ppm

The results obtained through the screening process showed that demulsifiers with higher volume of hydrophilic base chemicals will be more effective than demulsifiers with comparatively higher volume of hydrophobic base chemicals. From the Figures 3 a - c and at 20ppm, it was observed that A₁, A₂, A₃, A₄, A₅ and A₆ did better than A₇. However, A₃ and A₅ with ratios of 40/60 and 60/40 were more

effective in breaking this emulsion recovering 40ml of water for both cases after 40 minutes. At other dose rates of 30ppm and 40ppm, the formulations recorded good amount of water recovered with A₃ (40/60 hydrophobic/hydrophilic ratio) as the best, and observed decline in performance as the hydrophilic value increases culminating in a poor performance by A₇.

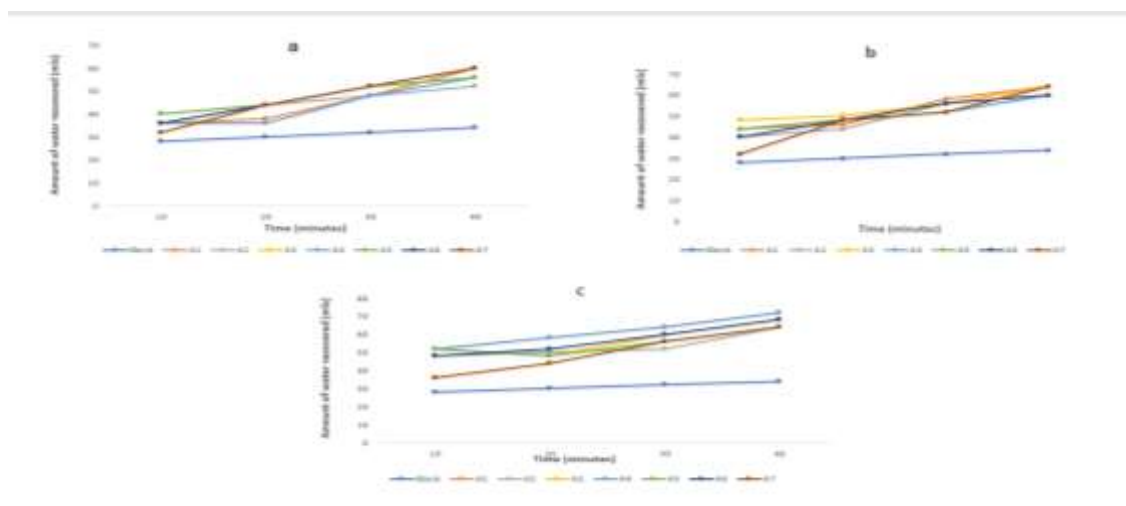


Figure 4 (a-c): The effect of hydrophobic/hydrophilic balance on performance of Demulsifiers A₁ to A₇ on crude D at (a) 20ppm, (b) 30ppm and (c) 40ppm

Figures 4 a - c shows the results of the effect of demulsifiers A₁ to A₇ formulation on crude D with rheological properties highlighted in Table 1 and at different dose rates with respect to the separation time.

The results obtained through the screening process like the other cases considered show that demulsifiers with higher volume of hydrophilic base chemicals were more effective than demulsifiers with higher

volume of hydrophobic base chemicals for crude oils within the range of rheological properties highlighted in Table 1. From Figures 4 a - c, it was observed that A₁ was more effective in breaking this emulsion removing 60ml of water at 20ppm, 64ml of water at 30ppm and 72ml of water at 40ppm after 40 minutes, followed by A₄ with A₇ having the least performance in breaking this emulsion recovering 60ml of water at 20ppm, 64ml of water at 30ppm and 64ml

of water at 40ppm after 40 minutes. Whereas the performance of the demulsifiers at 20ppm and 30ppm were within the same range, the water recovered at 40ppm was 72mls. This could be attributed to the high water cut of the crude and the relatively high interfacial tension causing the interface to be loose and easy to separate. This therefore requires higher dose rates and increased concentration of the hydrophilic component.

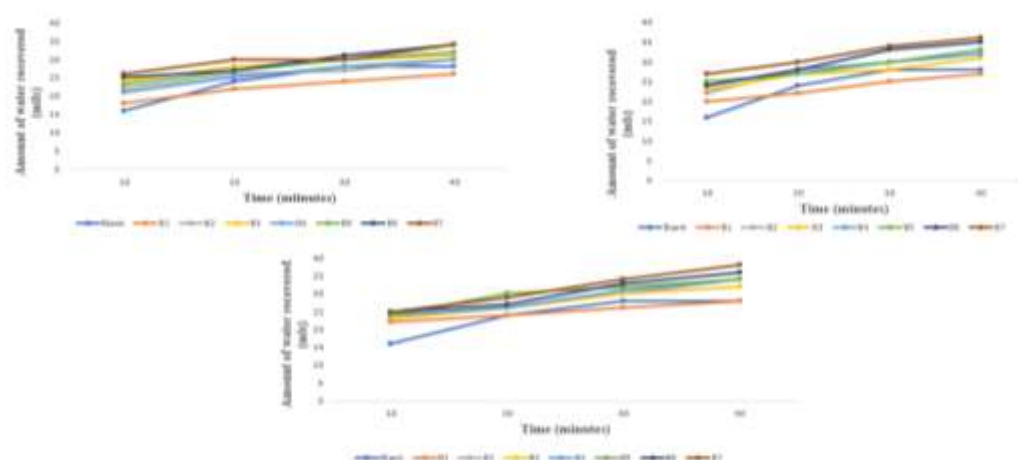


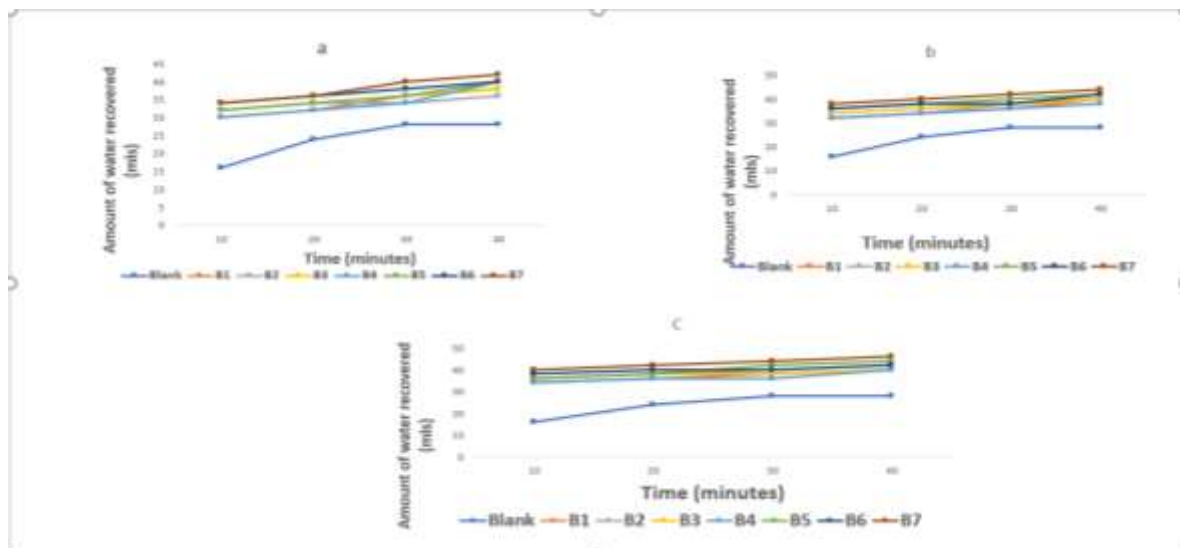
Figure 5 (a-c): The effect of hydrophobic/hydrophilic balance on performance of Demulsifier B₁ to B₇ on crude A at (a) 20ppm, (b) 30ppm and (c) 40ppm

Figures 5 a - c showed the results of the effect of demulsifiers B₁ to B₇ formulations on crude A with rheological properties highlighted in Table 1 and at dose rates of 20ppm, 30ppm and 40ppm with respect to the separation time. Unlike the cases discussed earlier, the application of demulsifiers B₁-B₇ on crude A, the results obtained showed that blends with higher ratio of hydrophilic ratio did perform better for crude with same range of rheological properties as crude A and B₇ gave the better result. Edith et. al. (2022) had reported that hydrophilic based demulsifiers are better suitable for water in

oil emulsions which is in tandem with this result, but at variance with what was concluded for the case of demulsifiers A₁-A₇ applied to crude A. This suggested that different demulsifiers will perform differently with different crude oil emulsions because of their different rheological properties, composition and chemistry. Therefore, for crude with rheological properties in the range of crude A, demulsifiers blend of Alkoxylated alkyl phenol formaldehyde resin (hydrophobic) with Amine-initiated polyol block copolymer (hydrophilic) should be hydrophilic rich and lean in hydrophobic.

This formulation will ensure a better performance as the result shows in this case with performance increasing progressively

from A₁ to A₇ and A₇ with the highest amount of water recovered for the different dose rates.



Figures 6 (a-c): The effect of demulsifier B formulation on crude B at (a) 20ppm (b) 30ppm and (c) 40ppm

Figures 6 a - c show the results of the effect of demulsifier B₁ to B₇ formulation on crude B with rheological properties highlighted in Table 1 and at dose rates of 20ppm, 30ppm and 40ppm with respect to the separation time. The results showed a progressive increase in performance from B₁ to B₇. While B₁ gave 38ml, 40ml and 40ml of recovered water for 20ppm, 30ppm and 40ppm respectively and at 40 minutes, B₇ gave 42ppm, 44ppm and 46ppm at 20ppm, 30ppm and 40ppm respectively after 40 minutes. Comparing the performance of the two demulsifier groups (A and B), for crude B, demulsifiers B₁-B₇ with higher volumes of hydrophilic chemicals did better unlike demulsifiers A₁-A₇ where the samples rich in hydrophobic components were better. These showed that demulsifier performances were specific to the crude oil emulsion. Therefore, the need

for this nature of information cannot be over-emphasized.

Figures 7 a-c showed the results of the effect of demulsifier B₁ to B₇ formulation on crude C with rheological properties highlighted in Table 1 and at dose rates of 20ppm, 30ppm and 40ppm with respect to the separation time. As observed for crude B, demulsifiers B₁ to B₇ performed better for formulations with higher hydrophilic composition when applied to crude C. This, as highlighted before, suggested that for demulsifier formulation based on Alkoxylated alkyl phenol formaldehyde resin (hydrophobic) with Amine-initiated polyol block copolymer (hydrophilic), the hydrophilic components should be more than the hydrophobic component. While it has been established that demulsifiers performances were crude specific, it is also

worthy to note that their chemical compositions were also crude oil dependent (Olusiji, et al., 2018). While particular class of demulsifier will do well in a field, it may

fail to replicate such sterling performance in another field. This variation is dependent of the rheological properties as well as the dominant emulsifying agents present.

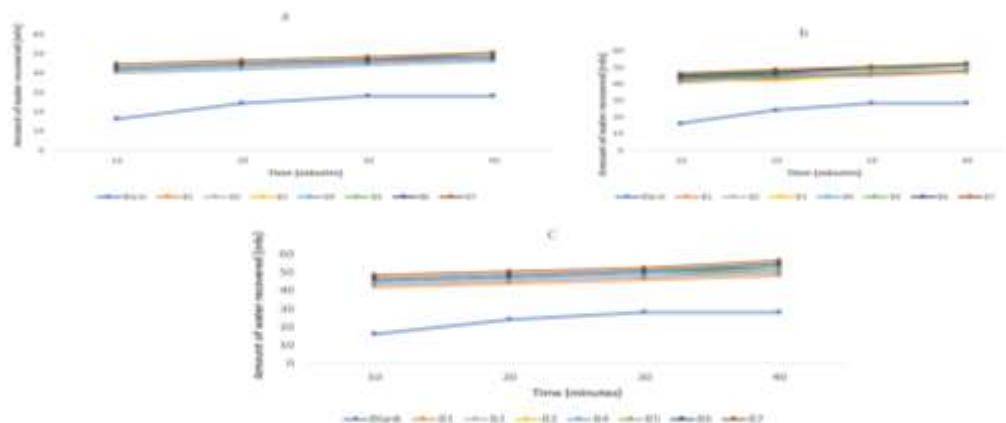


Figure 7 (a-c): The effect of hydrophobic/hydrophilic balance on performance of Demulsifier B on crude C, at (a) 20ppm, (b) 30ppm and (c) 40ppm

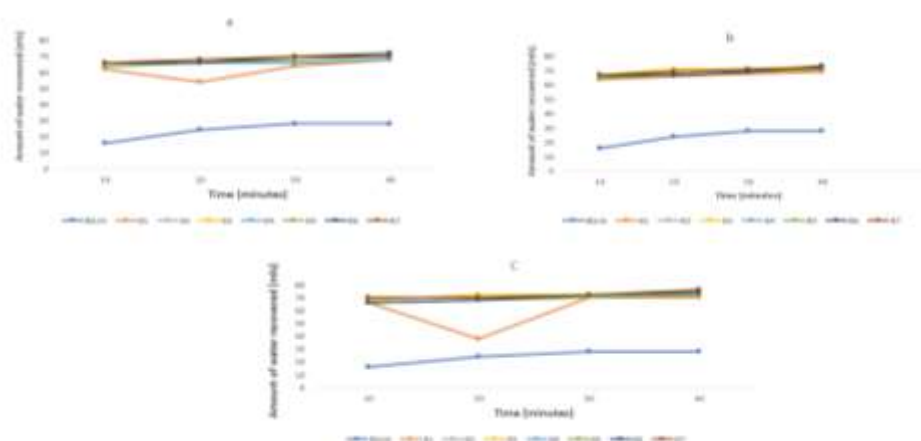


Figure 8 (a-c): The effect of hydrophobic/hydrophilic balance on performance of Demulsifier B on crude D at (a) 20ppm, (b) 30ppm and (c) 40ppm

Figures 8 a-c showed the results of the effect of demulsifier B₁ to B₇ formulation on crude D with rheological properties highlighted in Table 1 and at dose rates of 20ppm, 30ppm and 40ppm with respect to the separation time. Results obtained from the application of demulsifier B₁ to B₇ on crude D were in agreement with results obtained on application of demulsifier B₁ to B₇ on crude samples A, B and C. It was

observed here that at dosages of 20ppm, 30ppm and 40ppm for durations of 10 minutes, 20 minutes, 30 minutes and 40 minutes, the performances of the formulated demulsifiers improved showing a progressive water recoverability potential with increasing dose rates and time. It showed that for formulation based on Alkoxylated alkyl phenol formaldehyde resin (hydrophobic) with Amine-initiated

polyol block copolymer (hydrophilic), the amount of the hydrophilic component should be more than the hydrophobic component.

4. CONCLUSION

- Due to variations in properties of the crude, the demulsifiers formulated interacted differently with crude oil, which caused differences in the demulsification performance. A demulsifier formulation suitable for one type of crude might be unsuitable for another.
- Concentration of demulsifier affects their performance on the crude. It was observed that performance increased with increasing concentration up to the critical concentration.
- Demulsifier performance is a function of its hydrophobic and hydrophilic constituents. While in some cases, it requires high hydrophobic/hydrophilic ratio, in other cases it will require high hydrophilic/hydrophobic ratio
- For demulsifier formulation with epoxyl resin alkoxylate (hydrophobic) and propylene oxide/ethylene oxide polyol block copolymer (hydrophilic), it will require a high hydrophobic/hydrophilic ratio and for demulsifiers formulation with Alkoxylated alkyl phenol formaldehyde resin (hydrophobic) with Amine-initiated polyol block copolymer (hydrophilic), a high hydrophilic/hydrophobic ratio will be better.

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