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Experimental Assessment of Local Demulsifiers for Crude Oil Emulsion Breaking: A Study on *Jatropha curcas*, Indigenous Materials, and their Blends

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Abstract

Crude oil emulsion is one of the most challenging problems plaguing the phases of oil and gas production. Crude oil emulsion stability causes high viscosity of crude oil which leads to challenges in pipeline transportation and processing. This paper investigated the effectiveness of locally sourced material as a demulsifier, where the stability crude oil emulsion of varying viscosity by using three different formulated local demulsifiers such as *Jatropha curcas* extract, local materials, and their blends were investigated. The laboratory experiment was carried out using Crude oil I, II and III, blend of *Jatrophas curcas* and local materials into Sample A (100% *Jatropha curcas* extract), B (6:4), C (1:1), D (4:6), E (3:7), F (2:8), and G (100% local materials), at 60, 90, 120, 180, 240. 300, 360, 420 and 480 mins. The result generally showed that the lower the viscosity of crude oil the higher the water recovery (WR). Also, the demusifier blends had better WR when compared to without blends. Irrespective of the type of crude oil, demulsifier blends of equal ratio 1:1, 4:6, and 3:7 % led to maximum WR. For crude oil I, without delmusifier achieved 5.4 % WR, while Sample A had 86 % 77 % WR for Sample G at constant time of 480 mins, and 100 % WR for Sample D but at 36mins. Crude oil II, had WR of 4.0% at 480mins without demulsifier, Sample A, B, and D had water recovery of 79%, 72%, and 100% at 480 mins, respectively. Lower recoveries were observed for crude III majorly due to its higher viscous nature, as all samples had WR between 48.2 and 75%. This study has demonstrated that blend of locally formulated demulsifiers had better water separation capability.

Keywords: Crude oil emulsion, *Jathropha curcas*, local materials demulsifiers, Separation efficiency, water recovery

1. INTRODUCTION

There exist in an oil field's productive life when water co-production with hydrocarbons will reach an intolerable level. The water that coexists with the hydrocarbon in reservoirs slowly seeps into the formation's region where the hydrocarbons are produced. Regardless of the recovery process, water eventually enters the production (Ndubuisi et al 2023). In not so many instances, a field will generate water right away. Some reservoir wells that were drilled to deeper reservoir continue to produce water years later. The bottom portion of the well bore may occasionally be plugged back with cement, and intervals high in the formations may have holes drilled through them to limit the amount of water produced (Emuchay et al., 2013). During the production of crude oil which frequently includes water, a water-in-oil emulsion is created. Depending on the characteristics of the crude oil and, to some extent, the characteristics of the water, the stability of the emulsion varies from a few minutes to years (Mosayebi & Abedini, 2013).

The production of crude oil emulsions will continuously be a challenge to the oil industry (Sunil L and Saudi Aramco, 2007). Water-in-oil type emulsions also known as reverse emulsions, are typically created during the extraction and transportation of oil. Water in crude oil raises the cost of oil processing and transportation, consequently increasing the price of products from oil refinery (Faizullayev et al., (2022). Petroleum emulsions are typically stabilized through the development of a layer with elastic or viscous characteristics. This film is believed to be made up of a physical cross-linked network of asphaltenic molecules, which gather at the oil-water interface by lateral intermolecular pressures to form primary aggregates.

Demulsification can be carried out through three main methods: mechanical, electrical, and chemical. Among these, the most commonly used approach for separating water from oil in water-in-oil or oil-in-water emulsions is the addition of chemical agents known as demulsifiers (Oriji & Appah, 2012). The applications of these chemicals as demulsifiers for treating crude oil emulsions are specially tailored to act at the oil/water interface. Their high efficiency makes their use a very economical way and attractive to separate oil and water (Sulaiman et al. 2015). Several methods in use have suffered from drawbacks such as high costs of production and environmental concerns. The need to develop a cost effective and efficient demulsifier in treating crude oil emulsions without compromising quality and environmental safety is a major concern to the oil industry worldwide (Boisa et al 2021).

One of the most common techniques of emulsion treatment involve the addition of demulsifiers. These chemicals are modelled to counteract the stability impact of the emulsifying agents (Alao et al. 2021). Hence, chemical demulsification is one of the suitable methods in terms of both operational and economics. Among the chemical agents, interfacial active demulsifiers, which weaken the stabilizing films to enhance droplets coalescence, are preferred due to lower addition rates. However, these demulsifiers are costly and pose significant threat to the environment. It therefore becomes imperative to develop cheap and environmentally friendly demulsifiers.

Jatropha curcas is a non-edible plant widely distributed in tropical and subtropical regions. It has gained attention for its diverse industrial applications, especially in the formulation of eco-friendly additives used in petroleum operations. Therefore, the aim of this study is to evaluate the effectiveness of using a local demulsifier formulated from *Jatropha curcas* leaf extract, local materials, and their blend for breaking crude oil emulsion.

2.0 Materials and Methods

Materials used in this study include crude oil of varying viscosities, sodium chloride (for brine preparation), and demulsifier components such as *Jatropha curcas* extract and local materials (alum, native soap, and camphor). The *Jatropha curcas* leaves were harvested from Oleh, Delta State. Also, alum, camphor, and native soap were sourced from the community market in Oleh, Isoko South, Delta State, Nigeria. Different samples of Crude Oil (Crude oil I, Crude Oil II and Crude oil III) were collected from different locations in the Niger Delta area.

2.1 Extraction Process of *Jatropha* curcas

The *Jatropha curcas* leaves were rinsed with distilled water to remove surface impurities and air-dried at ambient temperature to eliminate residual moisture. Then the dried leaves were ground into a fine powder as shown in Figure 1, using a mechanical grinder. The powdered material was stored in an airtight container until extraction.

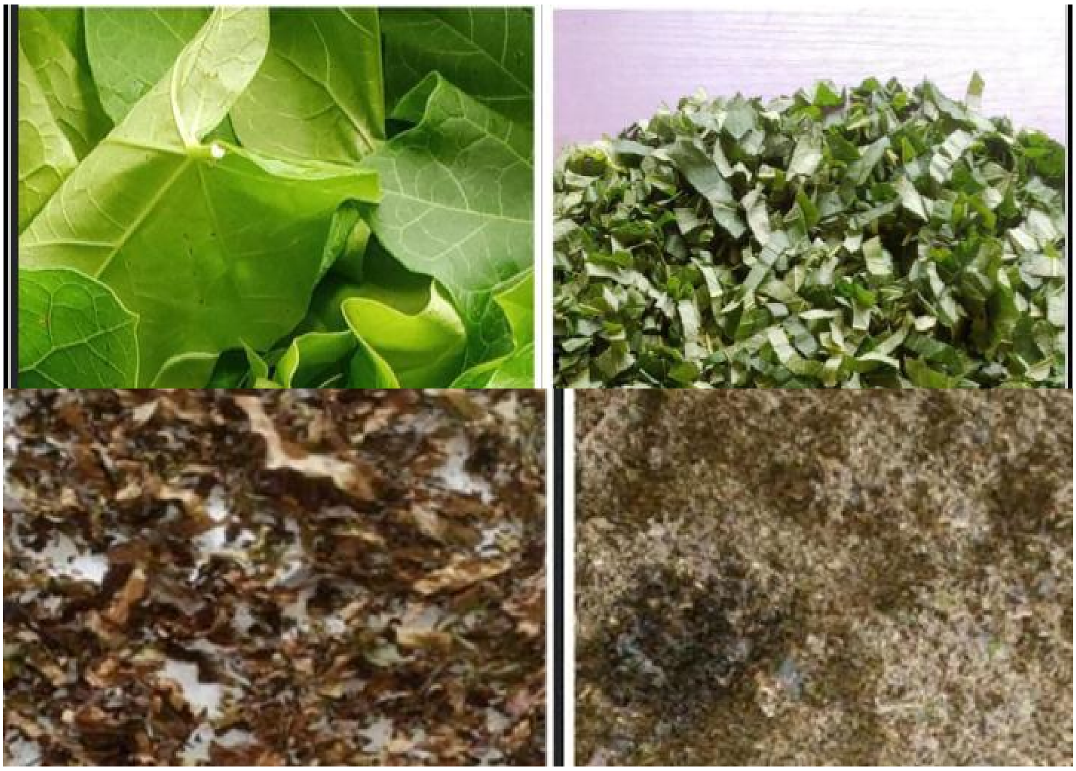

Figure 1 Pictorial view of fresh and dried Jathropha curcas leaves.

Extraction was carried out using a Soxhlet apparatus (shown in Figure 2) with ethanol as the solvent, selected for its effectiveness in extracting both polar and non-polar compounds. The procedure was conducted in accordance with the U.S. Environmental Protection Agency (EPA) Method 3540 for Soxhlet extraction (U.S. EPA, 1996). The extraction was performed at a controlled temperature of 90°C and continued until the siphoning solvent became clear, indicating complete extraction. The resultant extract was concentrated under reduced pressure using a rotary evaporator (RV10 V-C) at 40°C to remove residual solvent.

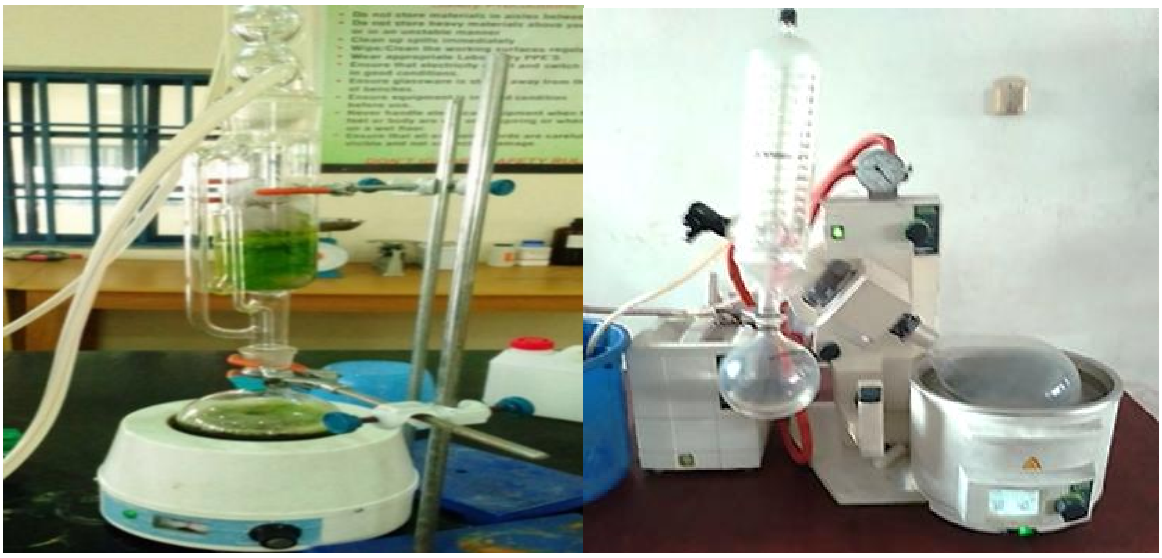

Figure 2: Soxhlet Extractor (BST/SXM-1) and Rotary Evaporator (RV10 V-C)

2.2 Production of local demulsifier

Local demulsifiers were formulated using a combined alum, native soap, and camphor. For each formulation, 100 mL of distilled water was measured into a 250 mL glass beaker. Then 10 g each of alum, camphor, and native soap were weighed and sequentially added to the beaker. Then the mixture was stirred continuously using a magnetic stirrer at 37°C until homogeneity. Then the resultant mixture was filtered using a standard-grade filter paper into a clean beaker to separate undissolved particles. The filtrate was collected and stored for subsequent application as a demulsifier. Then, five (5) blended formulations were prepared by varying the relative proportions of the clear filtrate and *Jatropha curcas* extract as shown in the Table 1.

Table 1 Composition of various demulsifiers

Blends	**Sample**	**DEMULSIFIER**	
		***Jatropha curcas* extract, %**	**Local materials, %**
1	A	100	0
2	B	60	40
3	C	50	50
4	D	40	60
5	E	30	70
6	F	20	80
7	G	0	100
-	Control	0	0

2.3 Preparation of Water in oil Emulsion

Water-in-oil emulsions were prepared using equal volumes of brine and crude oil, mixed with 1% emulsifier. The demulsifiers were prepared in various ratios (Table 1) and tested for their water-separating efficiency using the bottle test method at 60, 90, 120, 180, 240, 300, 360, 420 and 480 minutes.

2.4 Demulsification Process

In order to understand the efficacy of the formulated local demulsifier, different dosage of 4ml each of the demulsifiers was used and injected into 100ml of prepared water-in-oil emulsion. The volume of water (ml) removed or demulsified from the emulsion at different time interval was estimated using Equation (1). The pictorial view of the demulsification process is shown in Figure 3.

$$\text{Water Recovery, \%} = \frac{Volume\ of\ water\ recovered}{Total\ volume\ of\ water\ in\ emulsion} \times 100 \quad (1)$$

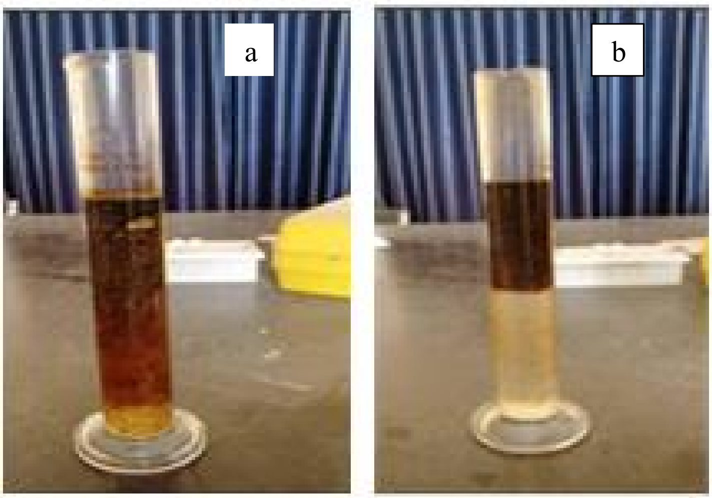

Figure 3 Demulsification process (a) Beginning of demulsification (b) 100% demulsified

2.5 Properties of Crude Oil

The viscosity of the different crude oil sample was calculated according to ASTM D-445, while specific gravity was determined according to ASTMD 1217-81. The properties of the different crude oil samples are shown in Table 2.

Table 2 Properties of crude oil I, Crude oil II and Crude oil III

Crude oil	Viscosity (mPa.s)	Specific gravity	API Gravity
Crude oil I	2.5	0.9213	22.09
Crude oil II	43.7	0.9520	17.13
Crude oil III	118.0	0.9544	16.76

3.0 Results and discussion

The percentage of water recovery from the various crude oil I, II and III used in preparing the various emulsions are shown in Figure 4, 5, and 6, respectively. Generally, the emulsion that was without a demulsifier showed that the emulsion was stable after 480 minutes (8 hours) with a 5.4% total volume of water separated.

3.1 **Effect of Demulsifiers on Water Separation Time**

The introduction of each of the demulsifies resulted in water separation from the emulsion. For Crude oil I, a low vicours oil, there were generally an increase in water recovery with an increase in time (60 to 360 min) among Sample A to E. However, further increase in time led to decrease in recovery.

As shown in Figure 4, Sample D exhibited best water recovery in the low viscosity crude oil. Sample D recorded 98% of water from the emulsion at 300 minutes (5 hours) and 100% water was recovered at 6 hours. Sample E was next to sample D in performance separating water from the emulsion. It took 420 minutes (7 hours) to break 100% of water from the emulsion. This implies that the blended demulsifier was effective in water separation from crude oil emulsion of low viscous crude oil sample. The use of sample A led to 86% water recovery at 480 mins (8 hours), while Sample G yielded 77% at same time which indicates a relatively low separation compared with the sample C. The presence of hydroxyl and carbonyl functional groups in *Jatropha curcas* extract, may support strong surface activity, therefore making *Jatropha curcas* extract a good demulsifier in comparison to the native ingredients demulsifiers. It can be seen that the lower the viscosity the faster the separation of emulsion. The performance of the demulsifiers is in agreement with Farah et al. (2005), whose result shows the percentage volume of water separated for three different crude oil samples by the new formulation of local demulsifier and their comparison with a commercially available demulsifier. Farah et al. reported that after 300 mins, the volume of water separated from the synthetic crude A, B and C water-in-oil emulsion are 51.7%, 49.2%, and 54.4%, while 63%, 60% and 66.2% volume were separated by the commercial demulsifier used.

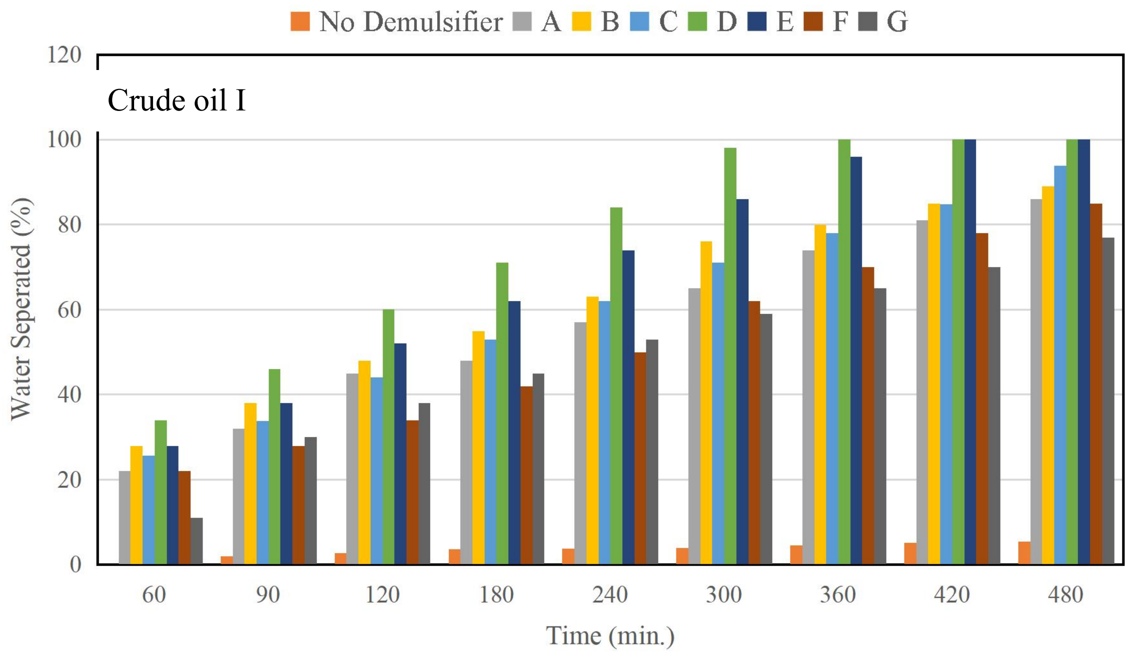

Figure 4: Percentage of water separated from the light viscosity crude oil emulsion (Crude oil I)

LEGEND	*Jatropha extract*	Native Ingredient	LEGEND	*Jatropha* extract	Native Ingredient
NO DEMULSIFIER	-	-	SAMPLE D	40%	60%
SAMPLE A	100%	-	SAMPLE E	30%	70%
SAMPLE B	60%	40%	SAMPLE F	20%	80%
SAMPLE C	50%	50%	SAMPLE G	-	100%

Figure 5 represent water recovery from an emulsion of a more viscous crude (Crude oil II) than in crude oil 1. As shown in Figure 5, the formulated demulsifiers showed similar trend as in the low viscous Crude oil I. Sample D also performed better when compared with other demulsifiers. At the same concentration of applied, sample D recovered 100% of water while sample E recovered 99%. It can also be seen from Figure 4 that sample A, B, C, F, and G possess similar water recovering ability at 300 minutes of demulsification. From 120 to 360 minutes, the rate of water recovery consistently increased across all the demulsifiers. This characteristic seems to be inherent in the emulsion itself as it was visibly seen in the control emulsion without a demulsifier under this time interval.

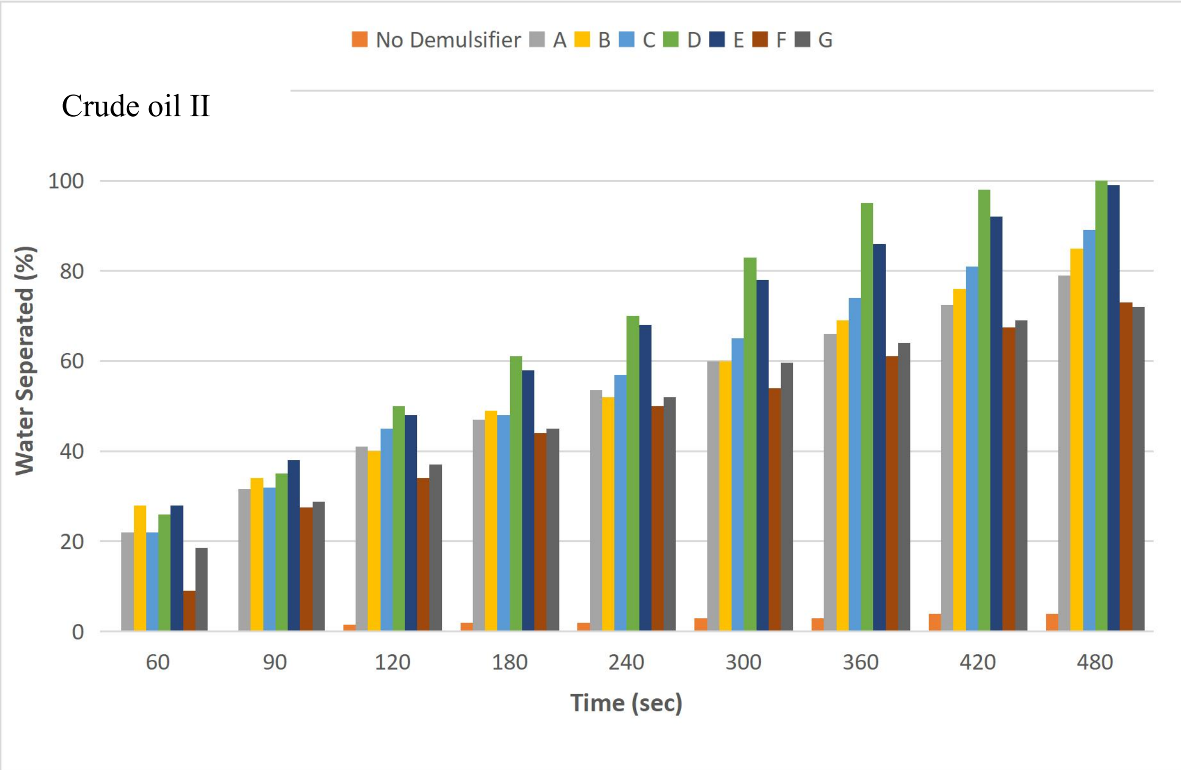

Figure 5 Percentage of water separated from the medium viscosity crude oil emulsion (Crude oil II)

LEGEND	*Jatropha extract*	Native Ingredient	**LEGEND**	*Jatropha extract*	Native Ingredient
NO DEMULSIFIER	-	-	SAMPLE D	40%	60%
SAMPLE A	100%	-	SAMPLE E	30%	70%
SAMPLE B	60%	40%	SAMPLE F	20%	80%
SAMPLE C	50%	50%	SAMPLE G	-	100%

The percentage of WR using Crude oil III is shown in Figure 6. Generally, the rate of recovery of water from the emulsion was lower when compared with those of Crude oil I and Crude oil II. In similar trend Sample D possesses highest ability to recover water from the emulsion with water recovery rate of 81%. This was followed by sample C, B, E, A, F and G with 75%, 71%, 66%, 62.6%, 55% and 49.2% respectively. In contrast to the increasing trend from 60 to 360 min and decrease in WR as observed with Crude oil I and II, there were generally increase in recovery rate from 60 to 480 mins for all Samples.

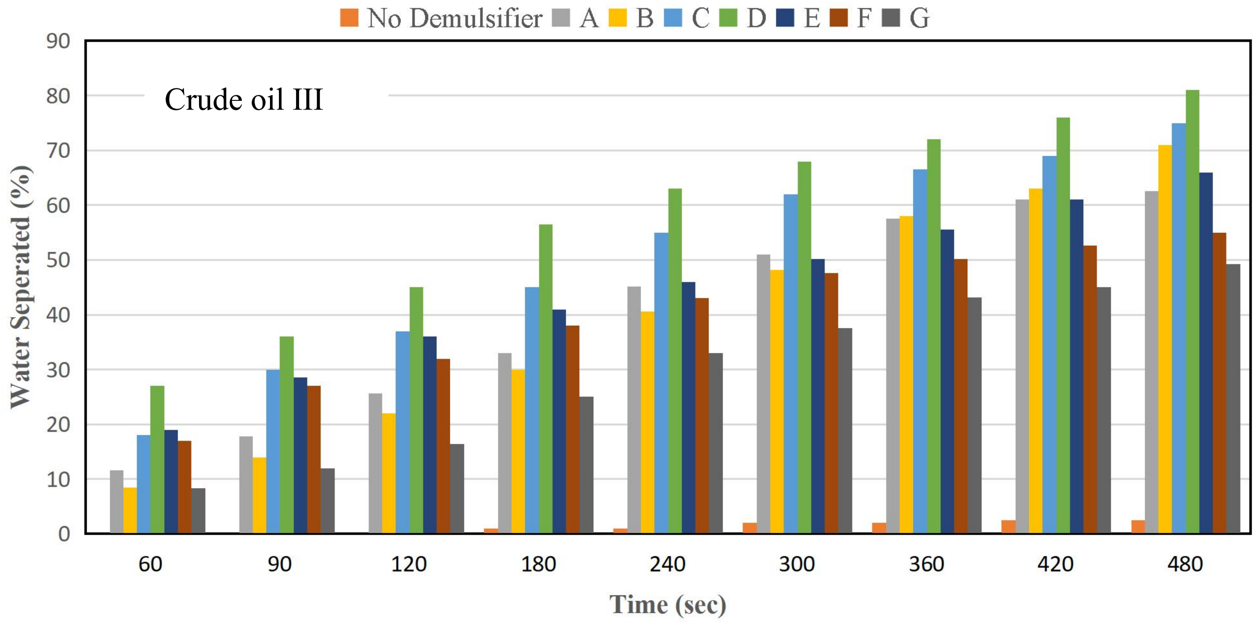

Figure 6: Percentage of water separated from the high viscosity crude oil emulsion (Crude oil III)

LEGEND	*Jatropha extract*	Native Ingredient	**LEGEND**	Jathropha extract	Native Ingredient
NO DEMULSIFIER	-	-	SAMPLE D	40%	60%
SAMPLE A	100%	-	SAMPLE E	30%	70%
SAMPLE B	60%	40%	SAMPLE F	20%	80%
SAMPLE C	50%	50%	SAMPLE G	-	100%

3.3 Effects of Demulsifiers on Viscosity of the Emulsion

This section is on the influence of three formulated demulsifiers on the stability of emulsion and how it impacts the resistance of fluid flow. The emulsion was studied for up to 480 minutes as shown in Figure 7 to 9. As shown in Figure 7, light viscosity emulsions (that is low-viscosity crude oil) were less stable and more easily separated, making demulsification faster and more effective. Sample D again shows the most effective viscosity reduction, reducing from 2.5 to 1.0 mPa·s by 480 minutes. This reflects the quick coalescence and separation, while sample A provides a consistent but moderate reduction to 1.6 mPa·s, effective for basic demulsification needs and Sample G has the least impact, ending at 1.8 mPa·s, showing lower or partial emulsion resolution. The low starting viscosity aids all demulsifiers, but synergistic action sample D remains superior in speed and completeness of separation.

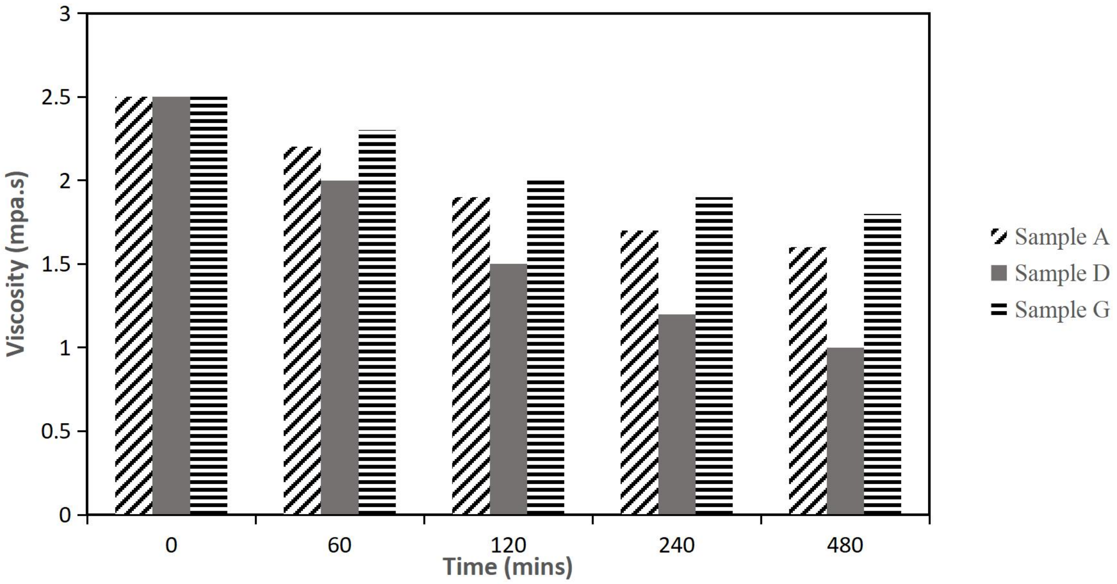

Figure 7 Effect of demulsifiers on Light viscosity emulsions sample

Moreover, as shown in Figure 8 is the medium-viscosity emulsions (43.7 mPa·s) representing a common field condition where emulsions were stable but still manageable with effective demulsifiers. Sample D led to the steepest viscosity decline, ending at 24.0 mPa·s, indicating rapid destabilization of emulsifying films and efficient water removal. Sample A performed moderately, reducing viscosity to 34.5 mPa·s, showing its utility as a standalone natural demulsifier. Sample G had the least impact, ending at 35.0 mPa·s, suggesting lower interfacial activity compared to the other agents.

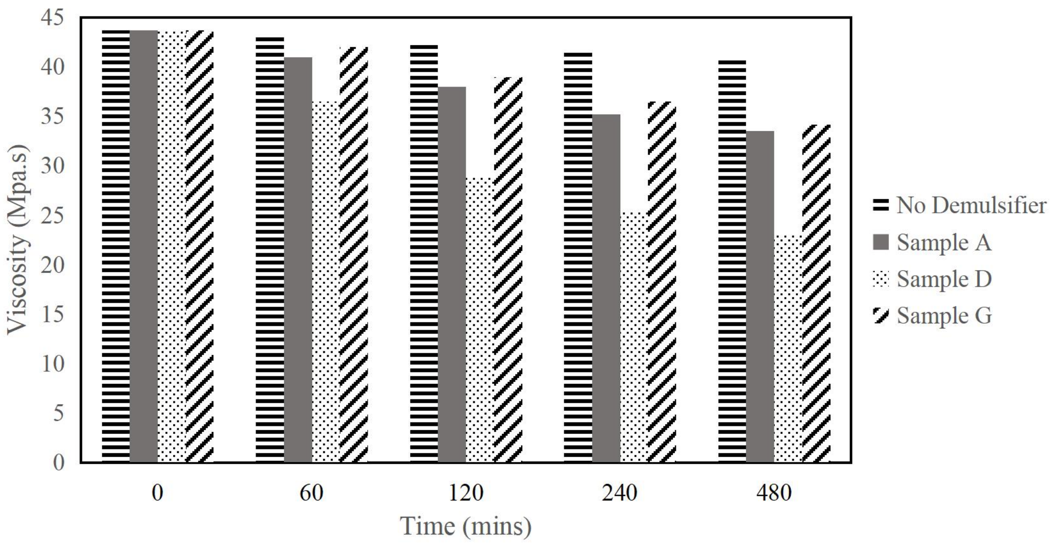

Figure 8 Effect of demulsifiers on medium viscosity emulsions sample

High viscosity emulsions present more resistance to demulsification due to stronger interfacial films, higher internal friction and more stable water droplet dispersions. Sample D as seen in Figure 9 showed the steepest reduction, from 118.0 to 70.0 mPa·s, demonstrating excellent performance under high viscosity. Sample A performed moderately, reducing viscosity to 92.0 mPa·s, indicating partial film destabilization while sample G had the least impact, dropping viscosity only to 94.5 mPa·s, reflecting its milder surface activity.

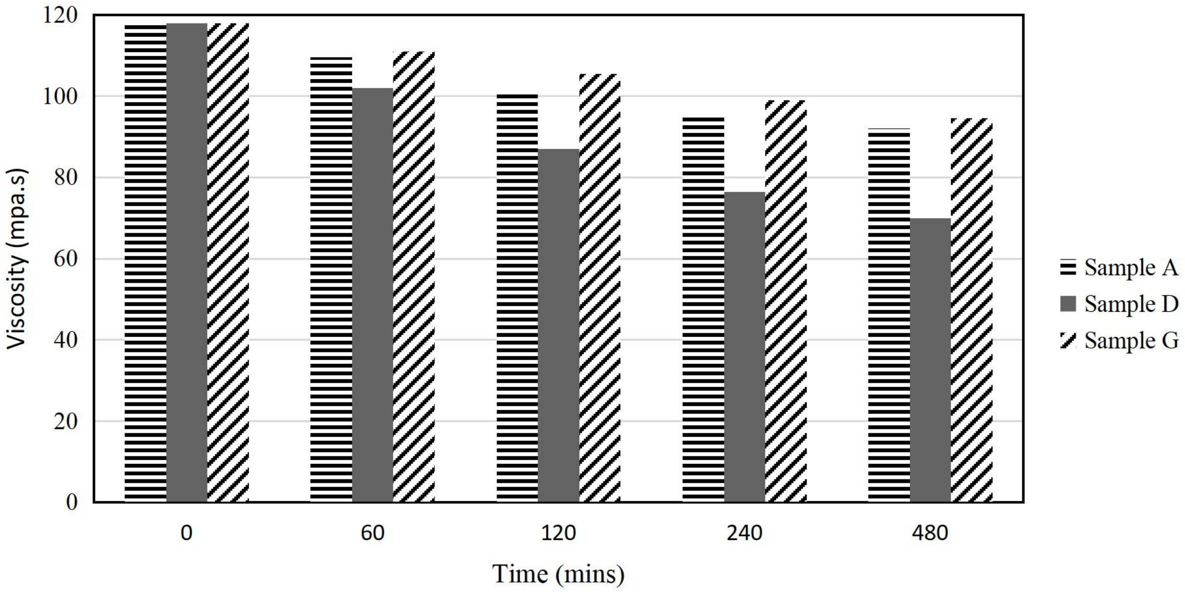

Figure 9 Effect of demulsifiers on Heavy viscosity emulsions sample

4.0 Conclusion

This study has shown that demulsifiers significantly affected emulsion viscosity by facilitating water droplet coalescence and reducing the internal phase volume. The extent and speed of this viscosity reduction depend on crude oil viscosity and demulsifier composition. Thus, changes in viscosity may not only lead emulsion resolution but also a useful metric for evaluating demulsifier effectiveness. Also, the locally sourced demulsifiers, especially the blend of *local* materials, have proven effective for crude oil emulsion breaking. They demonstrated high efficiency in water recovery, where Sample D consistently showed superior performance across all viscosity grades, making it a viable and environmentally safer alternative to synthetic demulsifiers.

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