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(RESEARCH ARTICLE)

Demulsification efficiency of the leave extract from African blackwood (D*elbergia melanoxylon)*

IWUOHA GN and CHIKWE TN *

Petroleum and Environmental Chemistry Research Group, University of Port Harcourt, Nigeria.

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Abstract

The demulsification efficiency of the leave extract from African Blackwood (Delbergia Melanoxylon) was determined using the bottle test method. Methanol, n-heptane and distilled water were used as extraction solvents respectively to extract the active ingredients of the leave. A 200 ml crude oil emulsion obtained from Niger Delta area Nigeria was introduced into six (6) calibrated 500 ml Teflon-stoppered bottles respectively, the leave extract in the solvent was introduced into each of the bottles as a demulsifier at concentrations of 50, 100, 150, 200 and 250 ppm with the sixth bottle left as control (without demulsifier). All the bottles were immersed in a water bath of 60 ^{0}C after mixing properly with a mechanical laboratory mixer and left for an hour. After an initial 5 minutes, the percentage of water separated in each bottle was recorded after 10 minutes. Bottle test was also carried out using a synthetic demulsifier (DXO77) on the same crude oil. Results obtained shows that the leave extract using methanol had the highest demulsification efficiency separating 50.5 % water from the crude after 60 minutes with a demulsifier concentration of 250 ppm. A 250 ppm of the synthetic demulsifier (DXO77) separated 51.0% of water from the crude after 60 minutes. The leave extract using n-heptane and distilled water separated 40.5 % and 32.5% water from the crude after 60 minutes at demulsifier concentrations of 250 ppm respectively. There is a consistency in the percentage of water from the crude within the last 15 minutes indicating that the separated water in the crude has reached its threshold, further increase in the demulsifier concentration at this point could lead to emulsification. The demulsification efficiency of plant extracts such as African Blackwood (Delbergia Melanoxylon) are influenced by the characteristics of the extraction solvent such as solvating power and extraction capacity.

Keywords: Demulsifier; Produced Water; Emulsion; Plant Extract; Extraction Solvent.

1. Introduction

Crude oil produced from older wells are accompanied with a great deal of water, most petroleum industries produce an average of three barrels of water for each barrel of oil especially from depleting reservoirs. Produced water in crude oil are contaminated water trapped in the reservoir rock, brought up along with oil and gas during production [1]. Most of the non-hydrocarbon components in crude oil are accessed through the water in crude (produced water). Dissolved oxygen that aids the survival of aerobic bacteria and anaerobic sulphate reducing bacteria responsible for the souring of crude and corrosion of metallic parts are accessed through produced water. Carbon dioxide in water produces carbonic acid which is the major cause of corrosion in oil and gas installations [2]. Heavy metal ions in crude oil such as lead, cadmium, chromium, vanadium, mercury find greater expression and residence in the water in crude, the environmental impact of heavy metals in crude oil cannot be overemphasized [3]. High salinity in crude oil can also be traceable to the breakthrough of saline water in the reservoir rocks during crude oil production [4]. Apart from the presence of several non-hydrocarbon contaminants in crude oil through the water-in-crude, the presence of water dispersed in crude oil forms emulsions that can lead to several challenges in the production and transportation of crude oil. A combination of oil and water forms two immiscible liquids of emulsion which are further stabilized in the presence

* Corresponding author: Chikwe T.N.

of asphaltenes, resins, naphthenes, parrafins which serve as natural emulsifiers [5]. The separation of water from crude oil through demulsification is of utmost importance to save the oil and gas industry from eminent collapse [6]. Demulsification can be defined as the breaking of crude oil emulsion into oil and water phases to separate water from the crude. There are several demulsification techniques however the use of chemical additives such as demulsifiers to enhance emulsion breaking process is the most widely used technique [7]. Owing to the adverse effects of synthetic demulsifiers, the need to develop an environmentally friendly plant based demulsifier is very imperative [8]. Several researchers have used various plants such as cashew nut oil, local starch, coconut oil etc as demulsifiers however the leaves of African black wood (*delbergia melanoxylon)* were used for this study. *Dalbergia melanoxylon* is one of the plants classified under the *Dalbergia genus,* others include *Dalbergia nigra* known as Brazilian rosewood, *Dalbergia latifolia* known as East Indian rosewood, *Dalbergia sissoo* (North Indian rosewood), *Delbergia cearensis* (Brazillian Kingwood) etc. The closest to *Delbergia melanoxylon* is *Delbergia welwitschii* known as South African Blackwood.

Dalbergia melanoxylon is a small tree of 4-15 metre tall with greyish bark and spiny shoots with leaves that falls off at maturity especially during dry seasons, the flowers are white and produced in dense clusters. They are grown in the coastal regions of west and east Africa such as Tanzania, Mozambique, Nigeria, Kenya etc. They are used in making furniture, artifacts, building constructions etc. The leaves are medicinal in nature and other *Dalbergia* species have been used locally in palm oil / water separation [9]. The aim of this study is to determine the demulsification efficiency of the leave extract obtained from African black wood (*dalbergia melanoxylon)* on a typical crude oil from Niger Delta Area. The leaves of *dalbergia melanoxylon* is shown in figure 1.

Figure 1 *Dalbergia Melanoxylon* Leaves

2. Materials and Methods

2.1. Sample Collection and Preparation

Dalbergia Melanoxylon Leaves were obtained from a farmland in a community called Oko in Asaba Delta State Nigeria. The authenticity of the plant was verified by a taxonomist and then the leaves were air dried at room temperature for a month, ground into a powdered form using a grinder and stored under laboratory conditions. The ground leaves were passed through a Soxhlet extractor using ethanol, n-Heptane and distilled water as extraction solvents respectively. The ratio of the volume of extraction solvent to the weight of the leave was 5:1 using 200 ml solvent per 40 g of the powdered leaves. The extraction solvents were separated from the plant extract by evaporation using a thermostatic water bath. The natural demulsifier was formulated using butanol and the *Dalbergia Melanoxylon* Leave extract from the different solvent [10].

2.2. Determination of Basic Sediment and Water (BSW) of Crude Emulsion

The BSW of a crude emulsion obtained from Niger Delta Nigeria was determined using Rotanta 460R Petroleum centrifuge. The test was carried out to ascertain the rough volume of water in the crude. The crude emulsion contained in the 1-liter glass bottle was properly agitated to ensure homogeneity and 50 ml was quickly poured into two of each 100 ml centrifuge bottle, each of the bottles was filled to the 100 ml mark with xylene. The centrifuge bottles were shaken again and then introduced into the trunnion cups on opposite sides of the petroleum centrifuge to maintain balanced condition. The centrifuge bath was set at 60 ^{0}C with a minimum relative centrifugal force of 1500 rpm and started to spin for 15 minutes. The volumes of sediment, water and oil in each tube were read and recorded to the nearest 0.05 ml. [11]. The BSW was calculated using equation 1:

$$BSW(\%) = \frac{S}{V} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots..(1)$$

Where:

S = *Volume of Sediment*

V = *Volume of crude emulsion*

2.3. Bottle Test Method

A 200 ml crude oil emulsion obtained from Niger Delta area Nigeria was introduced into six (6) calibrated 500 ml Teflon-stoppered bottles with each bottle labelled according to the concentration of demulsifier introduced into the bottle. *Dalbergia Melanoxylon* Leave extract from ethanol solvent formulated in butanol was introduced to each bottle at concentrations of 50 ppm, 100 ppm, 150 ppm, 200 ppm and 250 ppm respectively, the sixth bottle was used as control. This was also repeated for the plant extract from n-Heptane and distilled water. Each of the bottles were properly agitated severally using a laboratory mechanical mixer to simulate the natural mixing of crude oil and demulsifier in an oil and gas flow station and then immersed in a thermostatic water bath at a temperature of 60 ^{0}C with water separation observed for the initial 5 mins and subsequently at every 10 mins interval for a duration of an hour [12].

The efficiency of the demulsifier was determined based on the rate of water separation as indicated from the percentage water separation as shown in equation 2.

$$Water\ seperation = \frac{n}{V} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots.(2)$$

Where: n = *Volume of separated water*

V = *Total volume of water in the emulsion in ml*

Table 1 Water separated from Crude Using Synthetic Demulsifier (DX077)

		Concentration (ppm) /% Water Separation				
Time (minutes)	(0 ppm)	50 ppm	100 ppm	150 ppm	200 ppm	250 ppm
5	0	6.5	9.6	15.5	23.0	27.9
15	0	8	13.6	20.3	27.5	37.0
25	0	9.8	17.5	26	35.8	41.1
35	0	13.9	21.7	31.4	44.0	45.6
45	0	15.8	24.5	37.0	46.3	50.7
60	0.02	16	24.9	37.0	46.5	51.0

Table 2 Water separated from Crude Using *Dalbergia Melanoxylon* Leave Extract with Ethanol as Extraction Solvent

	Concentration (ppm) / % Water Separation				
Time (minutes)	50 ppm	100 ppm	150 ppm	200 ppm	250 ppm
5	6.7	9.8	15.7	22.9	27.7
15	8.3	14.1	20.3	27.2	36.5
25	10.1	17.8	25.9	35.5	40.5

35	14.2	21.9	31.1	43.8	45.2
45	16.1	24.5	36.6	46	50.5
60	16.2	24.9	36.8	46.2	50.5

Table 3 Water separated from Crude Using *Dalbergia Melanoxylon* Leave Extract with n-Heptane as Extraction Solvent

	Concentration (ppm) / % Water Separation				
Time (minutes)	50 ppm	100 ppm	150 ppm	200 ppm	250 ppm
5	3.0	5.8	10.1	16.6	21.5
15	5.9	9.9	15.3	20.2	28.3
25	7.1	12.1	18.7	25.8	33.5
35	9.8	15.4	22.5	29.2	38.8
45	11.1	18.0	26.5	32.2	40.0
60	11.2	18.1	26.9	32.5	40.5

Table 4 Water separated from Crude Using *Dalbergia Melanoxylon* Leave Extract with Distilled Water as Extraction Solvent

	Concentration (ppm) / % Water Separation				
Time (minutes)	50 ppm	100 ppm	150 ppm	200 ppm	250 ppm
5	0.8	1.7	4.3	7.9	14.1
15	1.5	3.4	7.5	13.5	20.9
25	2.8	4.5	9.6	18.5	24.8
35	3.6	6.2	14.4	23.4	29.5
45	4.2	6.8	17.7	25.2	31.8
60	6.1	7.1	18	25.3	32.5

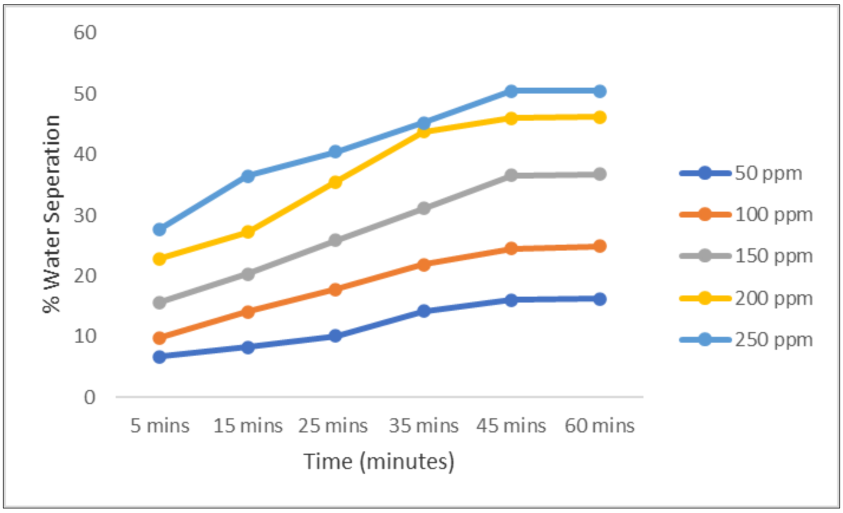

Figure 1 % Water Separation Versus Time (minutes) with Ethanol Leave Extract Concentration

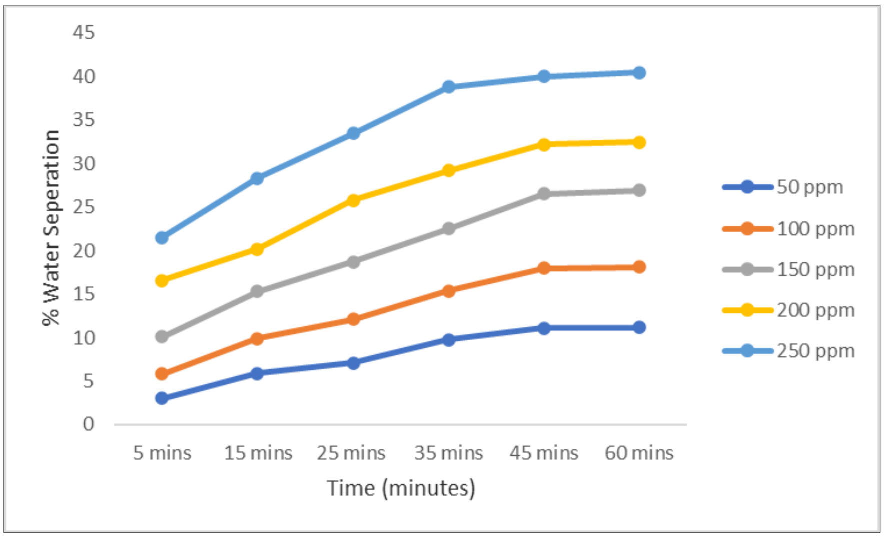

Figure 2 % Water Separation Versus Time (minutes) with n-Heptane Leave Extract Concentration

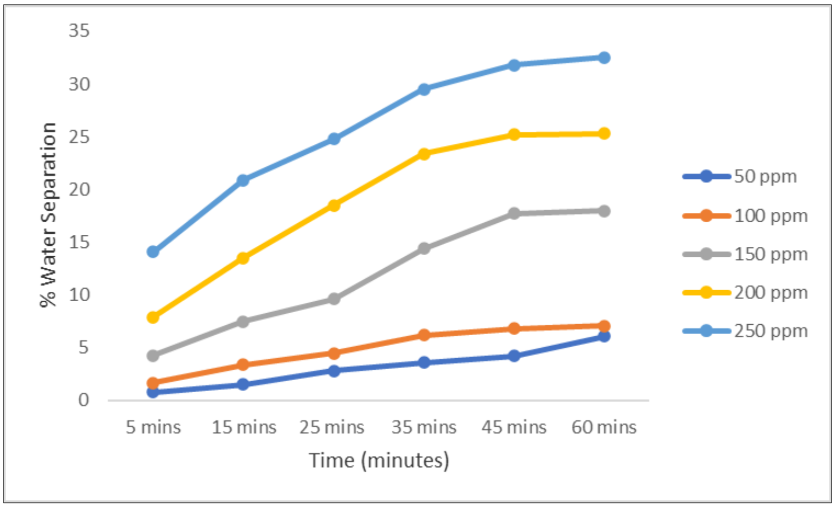

Figure 3 % Water Separation Versus Time (minutes) with Distilled Water Leave Extract Concentration

3. Discussion

The active ingredients of a demulsifier obtained from an organic source such as a plant determines its demulsification efficiency and this is highly dependent on the chemical constituent of the solvent used in extracting the demulsifying agent from its source. Table 1 shows the % concentration of water separated from a crude sample with no demulsifier added as well as using different concentrations of a synthetic demulsifier (DX077) as a demulsifying agent for a period of an hour with results observed at 10 minutes intervals. Results obtained shows a very little water separated (0.02 %) on the crude sample without demulsifier after an hour, however treatment of the same crude with different concentrations of the same demulsifier shows various percentage of water dropping out of the crude depending on the concentration of the demulsifier and the contact time between the demulsifier and the crude. Results obtained shows that the percentage of water separated from the crude increases with increase in demulsifier concentration as well as the contact time between the demulsifier and the crude sample with a consistency of percentage water separation observed at the highest demulsifier concentration of 250 ppm between 45 to 60minutes. Table 2 shows the percentage water concentration in the same crude but using *Dalbergia Melanoxylon* Leave Extract as a demulsifying agent and ethanol as an extraction solvent. Results obtained shows a very close (almost similar) percentage water concentration in the crude as with the synthetic demulsifier (DX077) in Table 1. However Results from Table 3 where *Dalbergia Melanoxylon* Leave Extract obtained using n-heptane as an extraction solvent shows a reduced percentage water concentration dropping from the same crude within the same time intervals as Tables 1 and 2. The percentage water concentration separated from the same crude further reduced in Table 4 when distilled water was used as an extraction solvent for *Dalbergia Melanoxylon* Leave Extract [13]. The demulsification efficiency of demulsifiers is dependent a great deal on their ability to break emulsions by migrating into water-in-oil interface, this is achieved by weakening the rigid films and improving water droplet coalescence. Alcohols are very good surface-active agents hence can promote good water separation, the polar water molecules are attracted to the hydroxyl group by hydrogen bonding between the hydrogen of water molecules and the oxygen in the alcohol (ethanol) [9]. Ethanol is a widely preferred extraction solvent for several reasons such as its extraction efficiency due to its high solvating power which enables it to extract all the active ingredients required. Ethanol also has a wide range of extraction selectivity as such can be used on a wide range substrate, it is more environmentally friendly than most organic solvents and affordable [8]. The use of n-heptane as an extraction solvent also has some advantages for instance n-heptane is immiscible in water therefore stays very dry after extraction as such can be easily reused, it recovers very fast and does not form azeotropes especially with terpenes however it does not have dewaxing abilities as such cannot be used in temperate regions with low temperatures except mixed with ethanol, this also explains why the demulsification capacity of *Dalbergia Melanoxylon* Leave Extract with n-heptane as an extraction solvent is not as high compared to when ethanol was used [14, 9]. The solvating power of ethanol increases its dewaxing efficiency which is very paramount in demulsification because most emulsions are formed from asphaltenes and naphthenates in crude which are sources of wax formation [10].

Comparing Tables 2, 3 and 4 shows that ethanol as an extraction solvent increased the demulsification capacity of *Dalbergia Melanoxylon* Leave Extract than n-heptane which in turn was a better extraction solvent than distilled water. Though distilled water can be used as an extraction solvent it does not however serve as an excellent extraction solvent for organic demulsifiers largely because of its immiscibility with oil which renders it surface inactive [8]. The graphs in figures 1, 2 and 3 shows a consistency in the percentage of water separated from the crude between 45 to 60 minutes, this indicates that the optimum water concentration in the crude has been separated, at this point further addition of demulsifiers could result in the reduction of the quantity of water separated from the same crude this is because good surfactants are also potential emulsifiers depending on the dosage applied [10].

4. Conclusion

The separation of water from crude oil is of critical importance in maintaining the desired characteristics and properties of the crude as well as its commercial value. The presence of water in crude oil negatively affects oil exploration and refining operations, therefore the use of an effective demulsifier in oil-water separation is essential. The leave extract from African wood (*Dalbergia Melanoxylon)* was very effective in separating water from crude oil having similar demulsification capacity as a synthetic demulsifier (DXO77). The demulsification capacity of *Dalbergia Melanoxylon* Leave Extract was influenced by the characteristics of the extraction solvent such as the extraction efficiency and solvating power. The leave extract by methanol had the highest demulsification capacity than the leave extract by n-heptane which in turn separated more water from the crude than the leave extract by distilled water. Demulsifiers from plant extracts such as *Dalbergia Melanoxylon* are more preferred than synthetic demulsifiers due to their environmental friendliness, availability and cost.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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