

Breaking of Oil -Water Emulsion for the Improvement of Oil Recovery Operations in the Niger Delta Oilfields

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ABSTRACT

Emulsified water is generally present in crude oil as a result of mixing occurring during production operations. The formation of emulsion leads to problems in production and also transportation. Therefore the need to break oil/water emulsions system through demulsification process using chemical surfactants for improved oil recovery operations. Selected cationic surfactants were effective in separating oil-water emulsions expected during a surfactant/polymer (SP) process for improved oil recovery. The aqueous phase of the emulsion contained an anionic surfactant blend, alcohol and partially hydrolyzed polyacrylamide. Brine composition was a suitable mixture of formation brine with brine from polymer drive. The crude oil had an API gravity of 47.2. Bottle tests were conducted at ambient temperature, which is near the reservoir temperature. Both oil and water phases of acceptable quality were obtained after settling upon addition of 200ppm of octytrimethylammonium bromide (C₈TAM) at ambient temperature. Microscopy image showed significant coalescence after only 1 minute in C₈TAM system as the cationic surfactant reduced electrostatic repulsion among drops and shifted system behavior towards the balanced state between hydrophilic and lipophilic effect well-known to reduce emulsion stability. The amount of cationic surfactant may be reduced by adding it simultaneously with anionic demulsifier resin.

Keywords: *Oil-water emulsion, Demulsification process, Surfactant/polymer process, Enhanced oil recovery*

1. INTRODUCTION

In the petroleum industry, emulsion of oil and water is one of many problems encountered (Swindle, Wang and Kuznik, 2010). These emulsions have wide spread occurrence in petroleum recovery. The evaluation of stability of emulsion systems and selection of optimal conditions for oil recovery process must be closely scrutinized to achieve effective separation (Poindexter, et al, 2003).

Crude oil is a continuous, multi-component solution of light and heavy hydro carbons which can be classified as asphaltenes, hetero atomic species, high molecular weight saturated alkanes and light gases. The light gases keep the heavy components in solution and the solubility of these components depends on the composition of the crude oil and the operating conditions of pressure and temperature during oil production. Crude oil is seldom produced alone because it generally is commingled with water. The water creates several problems and usually increases the unit cost of oil production. The produced water must be separated from the oil, treated, and disposed of properly. All these increase costs. Moreover, sellable crude oil must comply with certain product specifications, including the amount of basic sediment and water (BS&W) and salt, which means that the produced water must be separated from the oil to meet crude specifications. Emulsions occur

in almost all phases of oil production and processing such as inside reservoirs, wellbores, wellheads and also at wet-crude handling facilities and gas/oil separation plants; and during transportation through pipelines, crude storage, and petroleum processing (Kokal, and Al-Dokhi, 2007).

Selecting the best demulsifier for crude oil emulsion is very important as it will aid in the development of cost-effective separation of such an emulsion, providing an oil phase containing no more than 0.3-0.5% water by volume and an aqueous phase containing no more than 200 ppm oil, preferably 100 ppm, reducing treatment cost on emulsion resolution and generate revenue from the residual quantities of oil, help overcome pumping production encountered during the crude oil transportation and processing, increase the Net Present Value of the residual oil and minimize cost of treatment to maximize profit. The work will be strictly based on experimental treatment of emulsion form during by chemical enhanced oil recovery process. Recovering (i.e displacing) trapped oil will become increasing prevalent and important as assets age and producers want to recover their remaining reserves which in most cases are well in excess of what they have produced to date (i.e over the current life of the field) (Nguyen and Sadeghi, 2011).

Many analysts agree that around half the world's original proven oil reserves of around 2.2 trillion barrels of conventional oil have now been produced, with about 1

trillion remaining. Although estimates vary considerably, the fact is that producing the second half of the world's ultimate recovery of conventional oil will be more difficult than producing the first half. Oil production growth is forecast to decline within the next few years, as illustrated by Campbell-Laherrere; their predictions suggest that production will soon be unable to keep up with demand.

2. ENHANCED OIL RECOVERY

During the past 40 years, a variety of Enhanced Oil Recovery (EOR) methods have been developed and applied to mature and mostly depleted oil reservoirs (Fu and Mamora, 2010). These methods improve the efficiency of oil recovery compared with primary (pressure depletion) and secondary (water flooding) Oil methods. Each of these EOR methods are highly energy intensive.

Enhanced Oil Recovery (EOR) is a term applied to methods used for recovering oil from a petroleum reservoir beyond that recoverable by primary and secondary methods. Primarily recovery normally refers to production using the energy inherent in the reservoir from gas under pressure or a natural water drive. Secondary recovery usually refers to injection of water or water flooding. Thus, enhanced oil recovery is often synonymous with tertiary recovery, although sometimes Enhanced Oil Recovery methods can be used earlier in the sequence. In some older discussions, water flooding was considered as enhanced oil recovery, but now EOR is generally thought to follow water flooding.

These groups of methods; 1 – 3 are well established while 4 is under developmental study. We have total of eleven EOR methods grouped under four types:

- (1) Thermal recovery methods are cyclic steam stimulation, steamflooding, and insitu combustion.
- (2) Gas miscible recovery methods are cyclic carbon dioxide stimulation, carbon dioxide flooding, and nitrogen flooding.
- (3) Chemical flooding methods are polymer flooding, micellar-polymer flooding and alkaline flooding.
- (4) Microbial flooding methods include cyclic microbial recovery and microbial flooding.

3. CHEMICAL ENHANCED OIL RECOVERY METHOD

Due to a growth of petroleum demand and price, more and more planned or anticipated EOR projects will become reality. Chemical enhanced oil recovery (EOR) methods are expected to play a major role in future global

crude oil production (Watkins, 2009; Hirasaki et al, 2009). While recovering the oil can be a challenge, treating production that results from chemical EOR projects can be an even greater challenge (Poindexter, et al, 2003). During the application of chemical enhanced oil recovery (EOR) floods, breakthrough of the injection chemicals periodically occurs resulting in stable emulsions. It is generally not possible to predict when chemical breakthrough will occur and flood designs try to eliminate this unwanted event from occurring. Some Enhanced Oil Recovery recommendations use a combination of surfactant and polymer (SP), alkaline, surfactant and polymer (ASP), and variations on these two themes.

In the event that chemical breakthrough does occur, it is critical to have processes in place that can handle the ever changing conditions that evolve during the course of crude oil recovery. The emulsions from such floods can yield both stable water-in-oil (normal) and oil-in-water (reverse) emulsions particularly if the return of injection chemicals is higher than expected. Injection chemical packages often use surfactant-polymer (SP) or alkaline-surfactant-polymer (ASP) mixtures (Nguyen and Sadeghi, 2011). These chemical cocktails are injected into the formation, and by their very nature, facilitate the removal of trapped oil. Surfactants lower the crude oil water interfacial tension (IFT), polymers increase the viscosity of the water phase which raises the capillary number and alkali saponifies naturally occurring acids which can increase their water solubility and further reduce the IFT. All physical factors above that enhance oil recovery can also greatly contribute to the formation of very stable emulsions which are ultimately quite different from naturally occurring emulsions which are stabilized by asphaltenes and resins. Traditional demulsifiers are often not effective on emulsions created by chemical floods Efeovbokha, et al, (2010). Like any emulsion, these induced (i.e basically synthetic) EOR –based emulsions need to be resolved or broken. Producing dry oil (generally < 0.5 BS&W), and clean water are requirements that all producers must achieve (Kokal, 2005).

However, breaking dilute oil-in-water (O/W) emulsions produced during surfactant/polymer (SP) and alkaline/surfactant/polymer (ASP) processes for Enhanced Oil Recovery is important for process success. These emulsions can be very stable, for example, as discussed by Li et al, 2006 for ASP processes. Emulsions for SP processes may have continuous aqueous phases containing an anionic surfactant blend and high molecular-weight, partially hydrolyzed polyacrylamide.

Owing to dilution by mixing with reservoir brine and the polymer drive, surfactant concentrations vary during the process and may range from below 0.1 to perhaps 0.5% by weight, depending upon such factors as injection concentration, reservoir heterogeneity, and process

design. Polymer concentrations up to about 0.1% by weight may be expected. Total dissolved solids may be 1-5% by weight with some hardness. A cosolvent, such as an alcohol, may also be present in the EOR chemical blend. The surfactant is preferentially soluble in the aqueous phase for these conditions and solubilizes some oil. That is, the aqueous phase is an Oil/Water microemulsion. A typical process requirement is to develop a cost-effective separation of such an emulsion, providing an oil phase containing no more than 0.3-0.5% water by volume and an aqueous phase containing no more than 200 ppm oil, preferably 100 ppm (Bryan and Kantzas, 2009).

Much effort has been devoted to breaking crude oil-brine emulsions from normal production operations (Angle, 2001; Sjoblom et al, 2003; Kilpatrick, 2009; Watkins, 2009; Hirasaki et al, 2009). Emphasis has been on breaking brine-in-crude oil emulsions and, thereby, reducing the brine content of the oil phase to acceptable values. Asphaltene layers at the surfaces of the brine drops are the chief source of emulsion stability in most cases.

Many compounds have been proposed or used as demulsifiers. One type of widely used demulsifier capable of displacing significant portions of the asphaltene layers in such emulsions and promoting coalescence consists of ethoxylated and/or propoxylated alkylphenol formaldehyde resins with molecular weights of a few thousand daltons. Experiments have indicated that, for a given emulsion, there is an optimal ratio of hydrophilic and lipophilic properties for these non-ionic demulsifiers, which provides the best separation (Berger et al, 1987, Smith and Covatch, 1989, Paulis et al, 1997). This behavior is consistent with observations that both oil- and water-continuous emulsions stabilized by surfactants of low molecular weight are least stable near conditions where hydrophilic and lipophilic properties of the surfactant film are balanced and spontaneous curvature is zero.

Rondón et al (2007) perform experiment on breaking of water-in-oil emulsions formed during oil slicks or petroleum production operations. He stated that the natural surfactant molecules (principally asphaltenes) that are present in the crude oil are the cause of emulsion and these persistent emulsions may be broken by adding a suitable demulsifier at the proper concentration to attain a so-called optimum formulation at which the stability of the emulsion is minimum. In his report, the concentration of asphaltenes was varied by diluting the crude oil with a solvent such as cyclohexane, toluene, or mixtures of them. His experimental evidence showed that there exists some critical asphaltene concentration at which the interfacial zone seems to be saturated. Beyond this threshold, which is typically around 1000 ppm of asphaltenes, the demulsifier concentration necessary to attain the emulsion's quickest breaking is constant. Below it i.e.

when the crude is highly diluted with a solvent, the optimum demulsifier concentration is found to be proportional to the asphaltene concentration. He went further to establish a map of emulsion stability versus asphaltene and demulsifier concentrations which exhibits a typical pattern for different demulsifiers and diluents and contributes to improving the interpretation of the demulsifying action.

4. MATERIALS AND METHODS

The materials used for the experiment are crude oil, pure cationic surfactants of the type: Alkyltrimethylammoniumbromides (C_6-C_{18}), Alkyldimethylbenzylammoniumbromides ($C_{10}-C_{18}$), Polymer (partially hydrolyzed polyacrylamide, HPAM), Alcohol propoxylate sulphate and alkyl sulfonate surfactants, brine solution and Diethylene glycol monobutyl ether (DGBE) and Iso-butanol co-solvent.

4.1 Experimental Methods

The experimental methods is divided into three steps to generate raw data to show the effectiveness of the chemical demulsifiers listed above as capable of breaking oil/water emulsions for improved oil recovery operations.

4.2 The Properties of Crude Oil

The physical and chemical properties of crude oil used for the experiment such as API gravity, viscosity, the asphaltene content and the mass of KOH/g of oil were obtained and listed in table 1.

4.3 Preparation of Synthetic–Produced Brine

A 5-gallon bucket (19.08 litres) was used to prepare a solution of synthetic – produced brine by adding deionized water and brine solution. Then, polymer (partially hydrolyzed polyacrylamide, HPAM) was added. The solution was slowly stirred for about 30min so as not to form clumps commonly known as fisheyes. The mixing continues for about 1hour. Thereafter, the preweighed surfactants (0.05 wt%) of alkyl-trimethyl ammonium bromides (C_6-C_{18}), alkyldimethylbenzylammoniumbromides ($C_{10}-C_{18}$), were added with alcohol propoxylate sulphate and alkylsulfonate surfactants and the resulting solution was stirred with a rod at 8rpm for another 30 min for proper blending.

4.4 Emulsion Preparation

The emulsion was prepared in (177 ml) glass prescription bottles by mixing the produced water (synthetic – produced brine) with oil. The mixture of the brine solution and crude was done in the ratio of 7:3, 70 ml of synthetic – produced brine using prescription bottles marked at 100 ml levels. A funnel plastic beaker was used

to pour 30 ml of crude oil into each of the prescription bottle to the 100 ml mark. The prescription bottle samples of emulsions were placed on the Eberbach shakers on high speed for 10 min to mechanically mix the mixture together.

4.5 Demulsification Tests

The demulsification tests were conducted in graduated 177 ml glass prescription bottle to allow for rapid water drop readings. About 200 ppm of demulsifiers (chemicals for demulsification) Separol NF-36 was added on the basis of the total weight of emulsion about 50 ppm. The resulting mixture in the sample bottles was then placed on Eberbach shakers on high speed for a 240 shakes/min for 10 min after which they were removed and emulsion separation noticed in the emulsion samples were recorded at various time intervals and water drop readings were recorded in milliliters as a function of time. Water drop values were equally used to gauge emulsion stability. Water samples were withdrawn from the bottom of the bottles using a syringe and the oil concentrations in water were measured from the turbidity measurements to indicate the slug grindout number. After the water drop readings, the resolved or partially resolved oil from each bottle was analyzed for water content. Using a syringe with a blunt-tipped needle, a small portion of the oil (about 6 ml) was withdrawn off with the tip of the syringe which was set to 15-20 ml above the theoretical oil-water interface as determined by the slug grind out value (a procedure that measures the water content of the emulsion). The treated emulsion of oil of about 200 ppm was added to a graduated API centrifuge tube containing an equal volume of an aromatic solvent (Iso-butanol co-solvent), and the contents was shaken by hand. The centrifuge tubes were then centrifuged at high speed of 2700 rpm for 3 min. After centrifugation, the percent residual emulsion, typically referred to as basic sediment (BS), and the percent water (W) were noted for each bottle. After recording BS&W values, an alkylsulfonate surfactant (a chemical known to resolve the remaining emulsion) was added to completely knockout remaining water from the emulsion at a higher dosage of about 300 ppm to the centrifuge tube. Such chemicals are generally called “slugging or knockout chemicals” and are typically low-molecular-weight sulfonate-based materials. After slugging, the tube was again shaken and centrifuged as previously described. The basic sediment was therefore completely eliminated and only water remained in the bottom part of the. Lastly, the slug grindout number (a measure of the water content of the emulsion) was prepared as a percentage while smaller values of BS&W and slug indicate drier oil.

5. RESULTS AND DISCUSSIONS

The results of this work are presented in tables 1 to 4 and figures 1 to 5. The tables 1 to 4 shows the properties of crude oil used, slug grindout (BS&W), Oil content in

water and effects of the cationic demulsifier dosage and settling time on BS&W for emulsions respectively, while figures 4 and 5 shows the effects of the cationic demulsifier dosage and settling time on BS&W for emulsions and relationship between the oil concentration and settling time with and without a cationic demulsifier for emulsions respectively.

The cationic surfactants of the type alyltrimethylammonium bromide were used to determine the effect on water and oil separation of produced emulsions resulting from surfactant-polymer floods. A clear separation of aqueous phase was found upon addition of around 200 ppm n-octyltrimethylammonium bromide (C₈TAB). The addition of this demulsifier to the produced brine did not appear to produce any precipitation, even when heated to about 40 °C. Precipitation can occur in mixtures of cationic and ionic surfactants. With 200 ppm of added C₈TAB, the cationic/anionic surfactant mole ratio is about 1:3, significantly lower than the 1:1 molar ratio where precipitation usually occurs. These surfactants are soluble in water but combine with anionic surfactant present in the brine of the initial emulsion to form ion pairs and reduce electric charge at the oil-water interfaces, thereby shifting phase behaviour of the oil/brine/surfactant system in the desired direction. C₈TAB seemed particularly effective at concentrations of 200 and 300 ppm. It produced clear brine phases (by visual inspection) after several hours and reduced water content in the oil to 0.8 per cent by volume after 2.75 h as shown in figure 1 below.

Preliminary Experiments, 18 Hours Equilibration

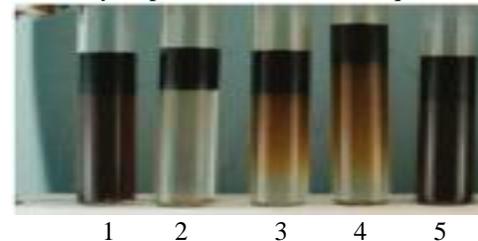


Figure1. Preliminary screening experiments for emulsions with 30% oil cut at ambient temperature

1 - 200ppm EO/PO Demulsifier A; 2 - 200ppm C₈TAB; 3 - 100ppm C₈TAB; 4 - 2000ppm C₈TAB; 5 - 200ppm Cationic Demulsifier V

Results of the bottle tests of figure 2 shown below indicate that the clearest aqueous phases were obtained with the combination of 50 ppm of demulsifier A and 50 ppm C₈TAB, followed by 50 ppm demulsifier A with 30 ppm C₁₄TAB respectively. These combined systems all have improved clarity of aqueous phases compared to 200 ppm C₈TAB alone. The results indicate a basis at 50 ppm C₈TAB for effective demulsification when used with 50 ppm demulsifier A for this system.

Bottle Tests: Cationic Surfactants & Demulsifier A, 21 Hours Equilibration

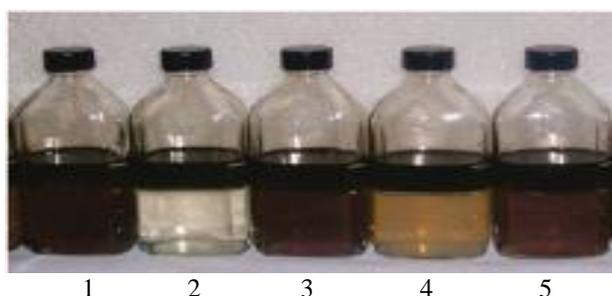


Figure 2. Separation produced by demulsifier A and different chain length of surfactant combinations after 21 hours for emulsions with 30% oil cut and ambient temperature

1 – No added chemicals; 2 – Demulsifier A + C₈TAB; 3 – Demulsifier A + C₁₂TAB; 4 – Demulsifier A + C₁₄TAB; 5 – Demulsifier A + C₁₆TAB

Similarly, microscopy studies showed large oil droplets separating from the emulsion shortly after the addition of C₈TAB, with larger droplets found at higher doses as shown in figure 3 below.

Microscopy of Emulsions after 1 Minute

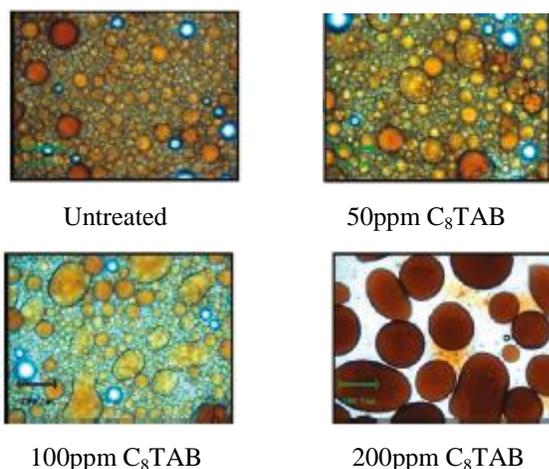


Figure 3. Microscopy of emulsions with 30% oil cut at ambient temperature

Table 1: Properties of Crude Oil

Properties	SP Oil
API (at 20 ⁰ C)	47.2 ⁰
Viscosity (at 20 ⁰ C, cP)	2.2
Specific gravity (S.G)	0.7918
Pour Point	3 ⁰ C
Wax, wt%	1.53
Sand content	15.4
BS&W % vol.	30.0

Table 2: Slug Grindout (BS&W) Percent

Emulsion	Grindout (BS&W) per cent
Untreated	30
Emulsion + C ₆ TAB	3.0
Emulsion + C ₈ TAB	0.4
Emulsion + C ₁₆ TAB	1.0

From the analysis of table 2 above, the slug grindout number (a measure of the water content of the emulsion) greatly reduced from the initial 30% by volume to 0.4% by volume by addition of C₈TAB surfactant.

Table 3: Oil Content in Water (ppm)*

Emulsion	Oil Content in Water (ppm)*
Untreated	224
C ₆ TAB	172
C ₈ TAB	112
C ₁₆ TAB	204

From table 3 shown above, it was observed that the lowest value of oil content in water was obtained when C₈TAB was added to the emulsion showing the effectiveness of this surfactant at resolving emulsion problems.

Table 4: Effects of the Cationic Demulsifier Dosage on BS&W for emulsions with 30% oil cut at ambient temperature and 40 min settling time

Cationic Demulsifier Dosage (ppm)	BS&W (%)
Untreated	13.8
50	8.8
100	0.9
200	0.6
300	0.6

From the observation of table 4 above rapid separation did not occur below 200 ppm, which is consistent with the observation that aqueous phase had reduced clarity below that dosage level as shown in table 4. It was found that the value of BS&W decreased with an increase in demulsifier dosage and settling time. At a demulsifier dosage of 200 ppm, BS&W decreased from 13.8 (untreated) to 0.6 per cent after 40 min of settling time. Similarly, figure 4 shown below indicates the effect of dosage and settling time on BS&W and oil content of the water phase for the cationic demulsifier. The oil content was reduced from 550 to 70 ppm after 4 h. These results showed that oil-water separation was improved with the use of the demulsifier.

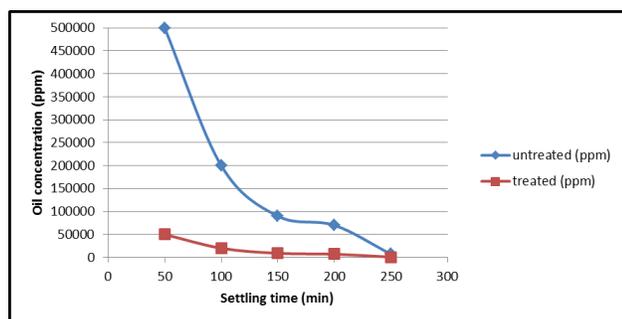


Figure 4. Relationship between the oil concentration and settling time with and without a cationic demulsifier for emulsion with 30% oil cut at ambient temperature

6. CONCLUSION

In this research work, the addition of 200 ppm of suitable surfactants accelerated the separation of oil-water emulsions representative of produced emulsions expected during chemical enhanced recovery operations and yielded oil and water phases with highly improved quality compared to the initial emulsions. Microscopy test confirmed that 200 ppm n-octyltrimethylammonium bromide (C_8TAB) produced significant coalescence shortly after it was added to the emulsions. This is in agreement with an increase of the oil droplet size in the presence of the demulsifier. While these results were obtained in connection with emulsion treatment studies specific for surfactants/polymer (SP) flooding, it is expected that the basic principle of using cationic surfactants demulsifier will be applicable to other similar processes employing anionic surfactants.

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