

Chemical Flooding Using Natural Recovery Agents at Reservoir Conditions

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Abstract - With rising global energy demand, increasing oil reserves by improving oil recovery at a reduced production cost is pertinent. Recent studies show that certain local materials can effectively act as chemical recovery agents; however, no extensive work has been conducted under reservoir conditions. This study aims at formulating an Alkaline-Surfactant (AS) blend for sandstone reservoirs using local materials at reservoir conditions by analysing the chemical compatibilities between the fluids, leading to an optimal low-cost Alkaline-Surfactant (AS) flooding. Two Alkalis: Potash, *Elaeis guineensis* ash and three surfactants: *Elaeis guineensis* oil, *Hibiscus X*, *Pennisetum purpureum* were evaluated in the laboratory with a formulated brine. Compatibility of aqueous phase was based on fluid-fluid interactions. Precipitation-free solutions were used for phase separation analysis which was then used to calculate optimal salinity. Sandstone core analysis was performed to determine the oil displacement efficiency of the individual local materials at temperatures (80°C) and pressures (6000psi). The best performing individual agents were used to form an AS blend and subjected to reservoir pressure (6000psi) and temperature (80°C). Results showed that all alkali systems and only two surfactant agents were compatible with brine. Type III microemulsion was obtained when alkaline and surfactant were combined forming the aqueous phase. Sandstone core flooding resulted in the formulation of an AS blend (2% Potash, 0.3% *Hibiscus X*, 0.2% *Pennisetum purpureum*) with an additional recovery of 10 % at reservoir conditions of 6000psi and 80°C. Results indicates that these local materials are capable of improving oil recovery even under harsh conditions.

Keywords: Surfactant flooding, Local materials, Alkaline flooding, Reservoir conditions, Displacement Efficiency

I. INTRODUCTION

The process of enhanced oil recovery using surfactants involves injecting surface active chemicals after water flooding into an oil reservoir in a bid to increase the capillary number (the ratio of capillary to viscous forces) thereby reducing the interfacial tension (IFT) between the displacing and displaced fluids. Experimental studies show that as the capillary number increases, the residual oil saturation decreases (Lake, 1989). The application of these chemicals is dependent on certain reservoir characteristics

such as reservoir type and depth, concentration of the formation brine and salinity; all of which ensures an effective oil displacement. Surfactant flooding involving the use of synthetic surfactants was successfully designed in the 1960s. However, due to the high adsorption rate and cost of synthetic surfactants, the concept of natural surfactants obtained from the reaction of alkali and crude oil was introduced. The addition of natural surfactants reduces the amount of synthetic surfactant adsorption onto the reservoir rock. The main recovery mechanism of surfactants (synthetic and natural) is its ability to lower interfacial tension (IFT) between the aqueous and oleic phases and also alter reservoir wettability towards being more water-wet. When the IFT is low, it annihilates the capillary forces that originally trap the residual oil thereby causing the oil to mobilize and flow. Liu *et al.*, 2008 observed that injecting natural surfactant could not attain optimal conditions needed to attain low IFT; however, injection of small amount of synthetic surfactant combined with the natural surfactant will achieve optimal salinity and an ultra-low interfacial tension thereby exhibiting a co-dependent mixing rule. Several reported pilot and field tests using Alkaline-Surfactant (AS) flooding such as the Big Sinking Field, Eastern Kentucky and Helper Field, Kansas, USA showed that only properly designed surfactant formulation would achieve high oil recovery. Phase behaviour analysis helps in selecting the best compatible chemical composition for a specific application and also in determining the optimum salinity of the alkaline-surfactant-brine system that can effectively enhance oil recovery. Furthermore, the use of conventional surfactants in certain conditions such as hard brines and high temperatures has been proven to be challenging (Tabary *et al.*, 2013) because surfactant adsorption increases significantly when hard

brine is used as the injection brine thus resulting in low surfactant performance, although (Novosad,1982) reported that surfactant adsorption should decrease with increasing temperature.

Interestingly, the recent use of certain local materials as surfactants for enhanced oil recovery has gained attention due to its low cost, low toxicity and availability. These local materials have been characterized and by their individual compositions, they are classified as local alkali, surfactant or polymer (Uzoho *et al.*, 2017). This has become necessary owing to the high cost of these imported chemicals (surfactants) which results in high production cost thereby minimising the profit margin of oil producing companies principally due to the reduced crude oil price. More so, the associated toxic effect of these chemicals has been proven to be harmful to the environment, aquatic and human lives. These local materials are considered to be a suitable replacement being that they are more readily available, less expensive and environmentally friendly. However, analysis of their phase behaviour is critical in ensuring the compatibility between the aqueous phase (alkaline-brine-surfactant system) and the oil phase as well as the optimum salinity which is the salinity wherein a surfactant splits into the microemulsion phase thereby achieving ultra-low IFT. This is true because phase behaviour of a surfactant required for the attainment of a low IFT is a function of the brine salinity. At higher salinity, greater than the optimum salinity (**over-optimised**), surfactant solubility is reduced in the brine causing the surfactant to partition into the oil phase increasing water microemulsion and gets trapped thereby resulting in surfactant loss and a Type II microemulsion. At intermediate (optimum) salinity, the microemulsion resides in the middle of the oil and water phase and is known as a Type III microemulsion. At lower salinity, less than the optimum salinity (**under-optimised**), surfactant partitions into the aqueous phase decreasing water microemulsion thereby leading to reduced oil mobilization and Winsor Type I microemulsion. This study seeks to design the right local Alkaline-Surfactant formulation and also shows how oil recovery using local

surfactants can be successfully applied in harsh conditions such as high temperature and pressure reservoirs.

II. BACKGROUND INFORMATION

Alkalis are strong water-soluble chemicals that produce hydroxide ions when dissolved in alcohol or water. Alkaline solutions have been used in enhanced oil recovery over the years due to its high pH value. During alkaline flooding, alkaline such as sodium carbonate is added to the injected brine, which increases the pH of the brine leading to the displacement of the crude oil. Alkali plays a unique role in the alkaline-surfactant flooding as the injected alkali can generate in-situ surfactants by its reaction with the organic acids in the crude oil. It also creates a more negatively charged rock surface due to higher hydroxyl ion concentrations which then gets adsorbed onto the rock surface thereby reducing the number of synthetic surfactants adsorbed. This improves surfactant efficiency and also reduces the production cost. The primary cause of surfactant adsorption is the interaction of electrostatic forces between the charged rock minerals and the charged hydrophilic surfactant group (Liu, 2007). Under low pH conditions, the interface is positive but under high pH, the interface will become negative. Another recovery mechanism of alkaline flooding is its ability to alter the wettability of the rock from oil wet to water wet. Wettability alteration will produce fluid redistribution in the pore space, which may be very beneficial for oil recovery (Morrow, 1990). In a normal condition, the reservoir is oil-wet occupying large pore spaces while the brine occupies the small pores. Alkaline creates a negative environment causing repulsion at the interface of the oil-brine thereby altering the wettability to water-wet. A basic requirement for alkaline flooding is that the crude oil in use must contain high acidic components, which will react favourably with the alkaline to produce natural surfactants. However, strong alkali at high concentration has some detrimental effects such as the formation of scales and emulsion in the production facilities, as such the addition of synthetic surfactants is required. Surfactants also referred to

as surface-active agents, are substances that adsorb onto the surfaces or interfaces of the system, altering the degree of surface or interfacial free energies. Interfacial free energy is the minimum amount of work required to create a unit area of the interface (Rosen, 2004). These wetting agents are amphiphilic in nature consisting of a hydrophilic (head) group and a lipophilic (tail) group in a single molecule. This implies that it has an insoluble non-polar hydrocarbon chain (tail) group attached to a polar water-soluble chain (head) group. This dual character allows surfactants to concentrate at a surface or interface and can alter surface properties. Generally, surfactants are categorised into four types based on the ionic charge of the hydrophilic group. They include Anionic (-ve), Cationic (+ve), Non-ionic (no ionic bond) and Amphoteric surfactants (both +ve and -ve). **Anionic surfactants** are the most widely used surfactant in chemical recovery due to their stable nature and low adsorption rate on sandstone reservoirs as opposed to carbonate reservoirs. More so, non-ionic surfactants are used as co-surfactants because of their high tolerance to salinity and their ability to improve the phase behaviour of the surfactant system (Sheng, 2013). The phase behaviour of a surfactant solution is largely affected by the salinity of the brine. Certain parameters are used to characterize surfactants. The most commonly used parameters are the Critical Micelle Concentration (CMC) and Solubilization Ratio. These two parameters are closely related to IFT and can be determined from the phase behaviour analysis of surfactants. CMC is an important surfactant characteristic and is defined as the lowest surfactant concentration above which micelles are automatically formed (Sheng, 2011). Micelles are aggregates of surfactant molecules consisting of 50 or more monomers in each aggregate. As the surfactant concentration increases and begins to form into micelles, the surface tension decreases. Above a certain concentration known as the CMC, any further increase in the surfactant concentration will only result in increasing the micelle concentration with little or no change in surface or interfacial tension. Solubilisation ratio which is a function of salinity is defined as the ratio of solubilized

water or oil volume to the volume of surfactant in the microemulsion phase. Solubilization ratio of water to surfactant, V_w/V_s , decreases as salinity increases, while Solubilization ratio of oil to surfactant V_o/V_s , increases as salinity increases. The point of intersection in a plot of oil and water solubilisation ratio against salinity is termed Optimum Salinity. Healy and Reed (1977) also reported that Optimal salinity plays a significant role especially in relation to interfacial tension and it is the salinity needed for maximum oil recovery during core flooding.

III. MATERIALS AND METHODOLOGY

Two local alkalis, two local surfactants agents and a bioethanol which acts as a non-ionic surfactant was used in this experimental study. The local surfactants used were *Elaeis guineensis* oil, *Hibiscus X*, a plant extract that contains mainly polyhydroxyl compounds and saponins and a non-ionic surfactant (bioethanol) obtained from *Pennisetum purpureum* (elephant grass plant). The local alkalis used were Potash and *Elaeis guineensis* ash. Ashes of *Elaeis guineensis* consisting mainly of Calcium, Potassium, Iron and Aluminium was sun-dried and then subjected to a temperature of about 550°C in a closed furnace. Potash was air-dried and pulverised. Isopropyl alcohol was used to extract alcohol from the *Pennisetum purpureum* plant. Extract of *Hibiscus X* was prepared using 10% potash solution. All these materials were locally sourced.

Chemicals used in the preparation of the brine were purchased from local suppliers. Synthetic brine was prepared in the laboratory to simulate the formation brine. The brine contained varying concentrations of sodium chloride and potassium chloride with total dissolved solids of 35,000ppm and a salinity of 3.5%. The crude oil used was supplied by Laser Engineering, Port Harcourt and was obtained from an oil field in the Niger Delta region of Nigeria with physical properties as shown in Table 1.

Table 1: Physical Properties of Crude Oil

Physical Properties	Values
Density @ 27°C	0.92 g/cm ³
API Gravity	21.5°
Viscosity	48.64 cP @ 30.5 °C
Colour	Brownish Black
pH	6.2
Total Acid Number	0.4 mg KOH/g

Cylindrical core plug with absolute porosity and permeability of 22% and 385 mD respectively was used for the core flooding analysis. The core plug was obtained from a clastic (sandstone) reservoir having lithology ranging from very fine to fine grain, well-sorted, moderately cemented to well-cemented core samples.

Experimental Procedures

Series of phase behaviour analysis were conducted to determine the compatibility of the various chemicals and the different phases at varying temperatures. The pH value (alkalinity) of the alkaline solution was determined. The critical micelle concentration of the anionic surfactants were also determined to ascertain the best concentration to use during oil displacement analysis.

Aqueous Stability Test

An aqueous stability or compatibility test between the alkalis and the brine solution was performed to determine the presence of any precipitation and/or cloudiness in the aqueous phase. Varying concentration (0.5%, 1.0%, 2.0%) of the selected alkali was mixed into a beaker containing 100ml of brine. The solution was well stirred using a magnetic stirrer and properly sealed to avoid evaporation. The solutions were subjected to higher temperatures of 50°C and 100°C and left for a period of 7days. Cloudy solutions containing solids such as precipitates were considered incompatible as only clear, cloudless and

compatible fluids were selected. Similar procedures were carried out on the two different surfactants in each brine to ascertain their compatibility.

Critical Micelle Concentration (CMC)

CMC is a principal characteristic of surfactants and can be determined from the physical properties of a surfactant such as electrical conductivity, surface tension, density etc. The CMC of the two local surfactants was determined to ensure the right concentration. The electrical conductivity of the surfactants was measured using a conductivity meter and a variation of conductivity values against surfactant concentration was plotted. The point of inflection on the plot was taken to be the Critical Micelle Concentration.

Phase Separation (Pipette) Test

Phase separation test was carried out primarily to determine the type of microemulsion formed and calculate the optimal salinity. Compatible alkali +brine and oil system were injected into an array of 5ml borosilicate pipettes. Each pipette contained equal volume of oil and alkali- brine solution at varying salinities. Samples were tightly sealed to avoid evaporation or inflow of oxygen and carefully inverted to allow a mix of the two phases. A second test was performed on compatible alkali + surfactant +brine and oil system. Lastly, a similar test was performed on alkali+ surfactant +co-solvent +brine and oil system. Fluid interfaces were recorded and samples in the pipette were observed under laboratory and reservoir temperatures of 100°C for an equilibration period of 21days for the presence of Type III microemulsion. Only solutions containing Type III microemulsion were selected because the presence of Type III microemulsion is indicative of an ultra-low interfacial tension. Level readings for aqueous, oleic and Type III microemulsion was recorded, their individual volumes, as well as oil and water solubilisation ratio versus salinity, was calculated in order to obtain optimal salinity.

IV. RESULTS AND DISCUSSIONS

The density and pH values of the two local alkali agents in distilled water was determined as shown in Table 2. As the concentration of the alkaline increases, the density, as well as the pH value increases with 2.0 wt.% Potash having the maximum value of 11.4 while 2.0 wt.% *Elaeis guineensis* ash has a lower pH value of 10.1. However, the two selected alkali sources are good candidates for alkaline flooding due to their high pH value (alkalinity) which is required in order to form an *insitu* surfactant through the process of saponification and alter wettability.

Aqueous Stability Test of Alkaline- Brine System

Aqueous stability test of local alkali, Potash in brine produced clear, compatible solutions at laboratory conditions and under elevated temperatures of 50°C and 100°C. Similar test performed on *Elaeis guineensis* ash also produced clear compatible solutions. This implies that the selected local alkalis have a high solubility and compatibility with the formulated brine even under high temperature.

Table 2: Density and pH values of Local Alkaline agents

Concentration	Potash		<i>Elaeis guineensis</i> ash	
	pH values	Density (g/cm ³)	pH values	Density (g/cm ³)
0.5	10.8	1.004	9.4	1.000
1.0	11.1	1.006	9.8	1.002
2.0	11.4	1.008	10.1	1.004

However, Potash was selected to be the alkaline source due to its higher pH value.

Critical Micelle Concentration (CMC) of Surfactant- Brine System

The Critical Micelle Concentration of the local surfactants in brine was determined. Figure 1 shows a plot of electrical conductivity values as a function of surfactant (*Hibiscus X*)

concentration. The point of intersection (0.3%) as seen in the plot is taken as the CMC. Table 3 shows the critical micelle concentrations of the local surfactants determined from a plot of surfactant concentration versus the measured electrical conductivity values of the surfactant. The result implies that with a CMC of 0.4% for *Elaeis guineensis* oil in brine, any further increase above 0.4 wt.% will result in little or no change in surface or interfacial tension. It also implies that a lower concentration of *Hibiscus X* in brine (0.3%) is needed compared to *Elaeis guineensis* oil with a CMC value of 0.4%. As reported by Adeniyi (2015) that a low value of CMC enhances surfactant mobility.

Having obtained the values of the Critical Micelle Concentration for each of the selected surfactant, aqueous stability test of the selected surfactants was performed to determine their compatibility at varying concentration and temperature.

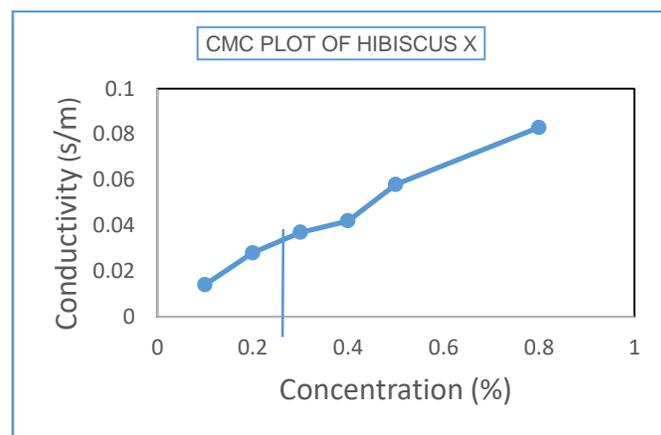


Figure 1: Plot of Critical Micelle Concentration of Surfactant, Hibiscus X.

Aqueous Stability Test of Surfactant- Brine System

Compatibility test of selected surfactants in brine was performed at laboratory and reservoir temperatures of 50°C and 100°C. The results as shown in Table 4 shows a high solubility and compatibility between *Hibiscus X*, *Pennisetum purpureum* and the formulated brine. However, in the case of *Elaeis guineensis* oil, solubility decreased as surfactant concentration increased both at laboratory and reservoir temperature of 100 °C. Based on the results

obtained, the use of *Elaeis guineensis* oil as a surfactant source was discontinued.

Table 3: Critical Micelle Concentration (CMC) of Local Surfactant Agents

SURFACTANT TYPE	CMC (%)
<i>Hibiscus X</i>	0.3
<i>Elaeis guineensis</i> oil	0.4

Table 4: Aqueous stability of Local Surfactant Agents

Conc. (%)	<i>Pennisetum purpureum</i>	<i>Hibiscus X</i>	<i>Elaeis guineensis</i> oil
0.1	Clear Solution	Clear Solution	Clear Solution
0.2	Clear Solution	Clear Solution	Clear Solution
0.3	Clear Solution	Clear Solution	Presence of Precipitates
0.4	Clear Solution	Clear Solution	Presence of Precipitates
0.5	Clear Solution	Clear Solution	Presence of Precipitates

Phase Separation Test of Alkaline- Brine-Oil System

Phase Separation (Pipette) test carried out on 2.0 wt.% Potash and brine at varying salinity with oil produced Type II microemulsion as such could not achieve optimum salinity. Similar analysis carried out on 2.0 wt.% *Elaeis guineensis* ash and brine solution at varying salinity with oil produced Type II microemulsion as such could not achieve optimum salinity as in earlier case. This further stresses the

fact that alkaline (in-situ surfactant) flooding alone cannot achieve optimum salinity.

Phase Separation Test of Surfactant- Brine-Oil System

Phase separation test conducted between the compatible surfactant in brine with oil resulted in Type I microemulsion or lower phase microemulsion where the microemulsion resides in the oil phase. This support the theory that surfactant flooding with the use of injected surfactant alone is unable to achieve optimum salinity.

Phase Separation Test of Alkaline- Surfactant- Brine-Oil System

Pipette test between clear aqueous solution comprising of 2 wt.% Potash + 0.3% *Hibiscus X* in varying brine concentrations and the oleic phase each having equal volume did not also produce Type III micro-emulsions under ambient and increased temperature of 100 °C; the solution produced a Type II microemulsion.

Phase Separation Test of Alkaline- Surfactant- Co-surfactant- Brine-Oil System

Pipette test between clear aqueous solution (0.3% *Hibiscus X*, 0.2 wt.% *Pennisetum purpureum*, 2 wt.% Potash) at an adjusted pH of 9 and medium crude each having equal volume produced Type III micro-emulsions under ambient and increased temperature of 50 °C and 100 °C.

Solubilisation ratio and optimal salinity calculations performed at room temperature equal to **2.7%**. Samples placed under a temperature of 100 °C for 7 days, produced Type III microemulsion and optimal salinity was calculated to be **3.0%** as shown in figure 2. It was observed that the addition of non-ionic surfactant (bioethanol) to the aqueous phase resulted in Type III microemulsion. The injection of synthetic surfactant to an alkaline solution as reported by Healy *et al.*, (1976) achieves optimum phase behaviour. The addition of the bio-ethanol (alcohol) as reported by Uzoho *et al.*, (2017), improves the solubility of the emulsion phase. It was further observed that an increase in temperature gave rise to a higher optimal salinity. This is

in-line with the findings of Healy *et al.*, (1976), where they reported that an increase in temperature increases the optimal salinity of an alkaline-surfactant system.

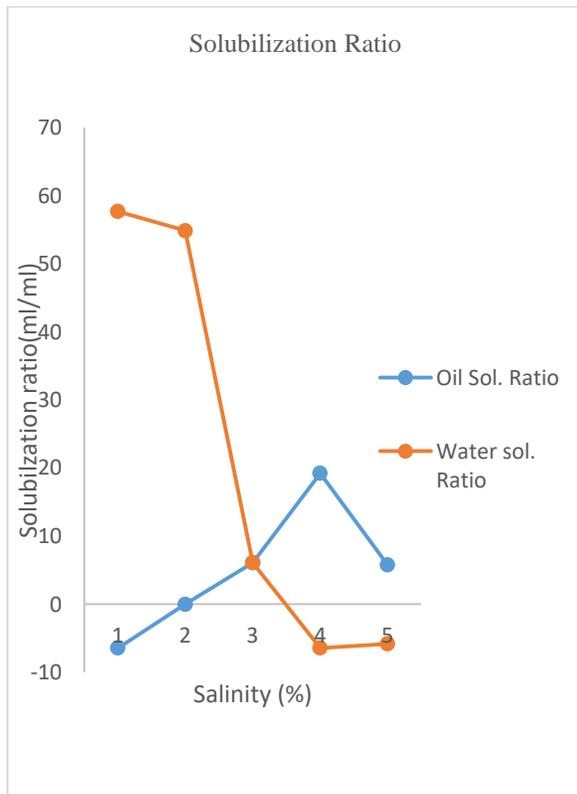


Figure 2: Solubilization Ratio of Potash- *Hibiscus X-Pennisetum purpureum* in Brine @ 100°C

Oil Displacement Analysis

Core flooding experiments were conducted to determine the effects of Alkaline- Surfactant (AS) flooding on sandstone reservoirs with a medium crude oil having an API gravity of 21.3 using unsteady state measurement where one phase is displaced by another.

All experiments were conducted at reservoir conditions of 6000 psi and varying temperatures of 50°C to 80 °C.

Properties of sandstone core samples used as the porous medium as well as crude oil properties are outlined in Tables 5 and 6 respectively. The results obtained for the initial water saturation, initial oil saturation and residual oil saturation after drainage and water flooding (imbibition) process at a maximum reservoir temperature of 80°C is

shown in Table 7. With an original oil in place (OOIP) of 11ml, a recovery factor of 63.6% was obtained after secondary recovery with brine leaving behind about 36.36% as residual oil saturation.

Table 5: Fluid Properties at Reservoir Temperature

Properties	Distilled water	Brine	Crude Oil	A-S
Density (g/cm ³)	0.9952	1.0167	0.9174	1.0013
S.G	0.9984	1.0199	0.9204	1.0045
°API	-	-	21.37	-
Dynamic Viscosity (cP)	0.87	0.91	50.2	0.91
pH value	5.5	5.9	6.2	9.8

Table 6: Rock Properties of the Core plug used for the EOR Study

PARAMETER	VALUE
LENGTH (cm)	5.8
DIAMETER (cm)	3.4
DRY WEIGHT (g)	128.5
WET WEIGHT (g)	143.2
PORE VOLUME (cm ³)	14.38
BULK VOLUME (cm ³)	52.66
CROSS SECTIONAL AREA (cm ³)	9.08
POROSITY (%)	27.3

Table 7: Calculated Values after Secondary flooding with Brine at 80°C

PARAMETER / UNITS	RESULTS AFTER IMBIBITION
Pore Volume (cm ³)	14.3819
Irreducible Water saturation at maximum oil saturation (cm ³)	3.3819
Initial water saturation (Swi) – %	23.51
Volume of crude in the plugs at Swi (cm ³)	11
Initial oil saturation (%)	76.49
Volume of crude recovered (cm ³) after imbibition	7
Volume of residual crude left over in the core (cm ³)	4
Residual oil saturation (Sor) – (%)	36.36

The selected Alkaline- Surfactant (AS) blend containing 2 wt.% Potash, 0.3 wt.% *Hibiscus X* and 0.2 wt.% *Pennisetum purpureum* was used as chemical enhanced oil recovery agent after secondary recovery (imbibition) and their displacement efficiency and recovery factor were determined as shown in Table 8.

Table 8: Displacement Efficiency of AS flooding at reservoir temperature

PARAMETER / UNITS	RESULTS AFTER IMBIBITION
Pore Volume (cm ³)	14.3819
OOIP (ml)	11
Oil recovered after imbibition(ml)	7
Residual oil volume	4
Displacement efficiency after imbibition (%)	63.64
Oil recovered after A-S flooding (ml)	1.1
Additional recovery after A-S flooding (%)	10
Displacement efficiency after A-S	27.5

flooding (%)

The results show an additional oil recovery of about 10% was obtained after the Alkaline- Surfactant flooding at a reservoir temperature and pressures of 80°C and 6000psi respectively.

V. CONCLUSIONS

- As alkaline concentration increases, pH increases, salinity decreases.
- At pH of 9, alkali (Potash) + surfactant (*Hibiscus X*) + brine solution resulted in ultra-low IFT.
- Local alkali and surfactant such as Potash and *Hibiscus X* are highly soluble even under reservoir temperature of 100°C.
- Local alkali, as in the case with synthetic alkali such as sodium hydroxide is unable to attain Type III microemulsion alone, a local surfactant is required.
- The presence of the bioethanol obtained from *Pennisetum purpureum* contributed to the hydrocarbon compatibility and emulsification process.
- *Hibiscus X* (0.3%) had a lower critical micelle concentration than that of *Elaeis guineensis* oil (0.5%).
- The effect of temperature was seen as an increase in temperature (100°C), increased the optimal salinity of the alkali and surfactant system.
- The AS blend containing 2 wt.% Potash, 0.3 wt.% *Hibiscus X* and 0.2 wt.% *Pennisetum purpureum* resulted in a displacement efficiency of 27.5% at reservoir conditions of 6000psi and 80°C.

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Declarations

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Conflicts of interest

The authors have no conflicts of interest to declare that is relevant to the content of this article

Availability of Data and Material

The data and materials used for this study are available

Code Availability

Not Applicable

Author Contributions

All authors contributed to this research study concept and design. Material preparation, data acquisition and analysis were performed by Obuebite Amalate Ann, Okwuonna Obumneme, Onyekonwu Mike and Akaranta Onyewuchi.

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