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Rheological characteristics of metal oxide nanocomposite and its application in enhanced oil recovery

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Abstract. A nanocomposite is a mixture of a nanoparticle and a complementary substance. In crude oil recovery, it is most commonly the combination of a nanoparticle and a polymer. In enhanced oil recovery, metal oxide nanoparticles may be combined with polymers and injected into a reservoir after water flooding to achieve greater recovery of crude oil; however, current research in this area is limited. In this work, rheological properties and oil recovery potential of two nanocomposites: alumina/xanthan gum and alumina/guar gum; were investigated. The viscosity of different concentrations of the nanocomposites was measured using the Model 800 OFITE Viscometer, and core flooding experiment was done using a Reservoir Permeability Tester. For both nanocomposites, it was observed that their viscosities increased with increasing concentration of alumina nanoparticles and the effect was higher as polymer concentration was increased. For the core flooding experiments, the xanthan gum and alumina composite achieved total recovery of 72.8 % while the guar gum alumina nanocomposite achieved total recovery of 69.3 %. The use of alumina combinations of nanocomposite is, therefore, promising and should be explored further.
Keywords: Nanocomposite, alumina, viscosity, polymer, oil recovery

1. Introduction

In recent years, the use of nanotechnology in different fields of science and engineering has gained great momentum. Through nanotechnology, novel materials having superior characteristics have been developed. Such characteristics include higher resistance to wear, higher strength, flexibility and more diverse applications [1]. Nanotechnology is a branch of science that develops and uses small materials that are on the scale of 1 to 100 nm. These small materials are called nanoparticles; they are smaller than what the eyes can see but bigger than an atom. Metallic nanoparticles such as aluminium oxide (alumina), copper oxide, iron oxide, nickel oxide, tin oxide, magnesium oxide zinc oxide and titanium oxide have chemical and physical properties that are different from their bulk metal form. These properties which include mechanical strength, high specific surface area, low melting point, specific magnetization and optic properties have proven to make metallic nanoparticles useful in diverse fields [2].

At the moment, nanotechnology has found application in almost all areas of manufacturing and production, including the petroleum industry, where it is used in oil mining, drilling, refining and

transportation [3]. It has also gained more prolific use because of the disappearance of "easy oil." Using conventional methods of oil recovery, only about 30 % of the original oil in place can be extracted after primary and secondary techniques [4]. It means that when oil is discovered at shallow depth (easy oil), about 70 % is wasted if only conventional techniques of oil recovery are applied; hence the need for enhanced oil recovery techniques. The oil industry has therefore looked to the use of nanoparticles as a potential solution to maximizing the recovery of easy oil [5, 6].

Different types of nanoparticles are being tested for use in enhanced oil recovery, the most common being aluminium, silicon and zinc nanoparticles. Nanoparticles enhance oil recovery by wettability alteration and the reduction of interfacial tension (IFT). The adsorption of nanoparticles on surfaces changes the wettability and surface energy of the nanoparticle, and the characteristic of the nanoparticle is controlled by the surface energy. Metallic oxides such as copper oxide, iron oxide and aluminium oxide have remarkable properties that give them a wide variety of applications in electronics, photonics, photovoltaics, catalytic conversions etc. The following reasons make nanoparticles viable options for use in enhanced oil recovery: magnetic separation can be used to separate nanoparticles from oil; they are inexpensive and ecologically friendly; the project cost for installation is low; they can move through the pore throat with reduced risk of impairment; and they have a high surface area to volume ratio [7, 8]. Some challenges affecting the use of nanoparticles in enhanced oil recovery include log jamming, mechanical entrapment, gravity settling and adsorption [9]. Log jamming occurs when the nanoparticles move at a rate slower than the displacing fluid, causing the nanoparticles to accumulate and block the pathway. Mechanical entrapment occurs when the size of the nanoparticle is bigger than the pore throat, causing an entrapment of the nanoparticle. Gravity settling occurs when the nanoparticles settle on the internal surface of the porous rock as a result of density differences between the fluid and moving nanoparticles [10, 11]. Adsorption occurs when the electrostatic forces on the internal surfaces of the reservoir formation attract the nanoparticles and cause them to be adsorbed to the surface. Brownian motion of the particles can also cause adsorption.

Nanoparticles can be mixed with a polymer to increase the stability of nanoparticles by mitigating some of the challenges associated with the use of nanoparticles. Nanoparticles in turn can increase the viscosity of the displacing fluid, thereby reducing fingering and increasing the overall efficiency of the oil recovery process. This mixture of nanoparticles and polymer is called nanocomposite, and its use has gained increasing interest among researchers. The effect of alumina nanoparticle has been investigated in the literature, and it was found to produce total recovery ranging from 37 to 46 % [12, 13]. In this work, rheological characteristics and the oil recovery potential of nanocomposites of alumina were investigated.

2. Experimental description

2.1. Materials

Sigma Aldrich alumina nanoparticle with the purity of 98 +% and an average size of 40-60 nm was purchased from a local dealer in Lagos, Nigeria. Xanthan gum and guar gum were purchased from a local dealer in Lagos, Nigeria. Other materials used include crude oil with API 24.8^0 gotten from the Niger Delta region of Nigeria, and core plugs acquired from Offshore Depobelt – Niger Delta.

2.2. Equipment

The OFITE[®] 800 Model Viscometer was used to measure the viscosity of all samples. The enhanced oil recovery process was initiated and actualized with the use of OFITE[®] Reservoir Permeability tester, and all

flooding was done at 28℃. The saturation of core plugs for porosity determination was done using Vinci Equipment at high-pressure.

2.3. Preparation of core plugs used

Core plugs were used for the core flooding equipment in the laboratory to measure the effects of testing fluids for this enhanced oil recovery process. The core plugs were cleaned in a soxhlet extractor using toluene as the cleaning solvent. Density and fluid viscosity were determined using a pycnometer and glass capillary viscometer, respectively. The core plugs were saturated using Vinci Equipment's high-pressure core saturator for Porosity Studies.

2.4. Preparation of nanocomposites

For each nanocomposite mixture, a plastic bottle was filled to 500 mL with deionized water. Respective quantities of aluminium and the required polymer (xanthan gum and guar gum) were then measured and added to the deionized water. For the viscosity measurements, five weight percentages of aluminium oxide nanoparticle were mixed with three different weight percentages of polymer to have 15 different combinations for each nanocomposite. For the core flooding experiments, three different weight percentages of aluminium oxide nanoparticle used were 0.1, 0.3 and 0.5; these three weight percentages of alumina were mixed with three weight percentages of polymer: 0.1, 0.3 and 0.5. To achieve nanocomposite of 0.1 wt% alumina and 0.3 wt% polymer, 0.5 g of alumina was added to 500 mL of deionized water, and then 1.5 g of polymer was added (Table 1).

Table 1: Composition of polymers and aluminium oxide nanoparticle

S/N	Polymer concentration [g,%]	Alumina concentration [g,%]
1	[0.5, 0.1] ~ XG1, GG1	[0.5, 0.1] ~ A1
2	[1.5, 0.3] ~ XG2, GG2	[1.5, 0.3] ~ A2
3	[2.5, 0.5] ~ XG3, GG3	[2.5, 0.5] ~ A3
4	[5.0, 1.0] ~ XG4, GG4	[5.0, 1.0] ~ A4

XG – xanthan gum, GG – guar gum

2.5. Viscosity measurements

Viscosity was measured using an OFITE 800 Model Viscometer. The mixture to be measured was filled into the sample cup of the viscometer. The rotor sleeve of the viscometer was immersed to the fill line of the sample cup. The viscometer was set to the required shear rate (10.21 s^{-1}) and the corresponding dial reading measurement was taken from the viscometer.

2.6. Core flooding

Core flooding using nanocomposites was carried out at 28^0C using an OFITE® Reservoir Permeability tester. The setup comprises of three different compartments that are either filled up with crude oil, brine or the nanocomposite solution. For regulation of the flow of fluid, each compartment is equipped with an inlet and outlet valves. The flood operates using a high precision pump to inject into one of the compartments at that instant, then acting as a driving force pushing a piston plate inside it, which drives the liquid contained

in the compartment at the same time isolating the driving fluid from the displacing reservoir fluid. The setup for the core flooding equipment (Figure 1) has the following parts: pump fluid, pump, valves, displacing reservoir fluid, piston to separate the oils, crude oil, brine, polymer (Treating fluid), pressure gauge, bypass valve, hassler cell holder with core, sleeve pressure, effluent into test tubes.
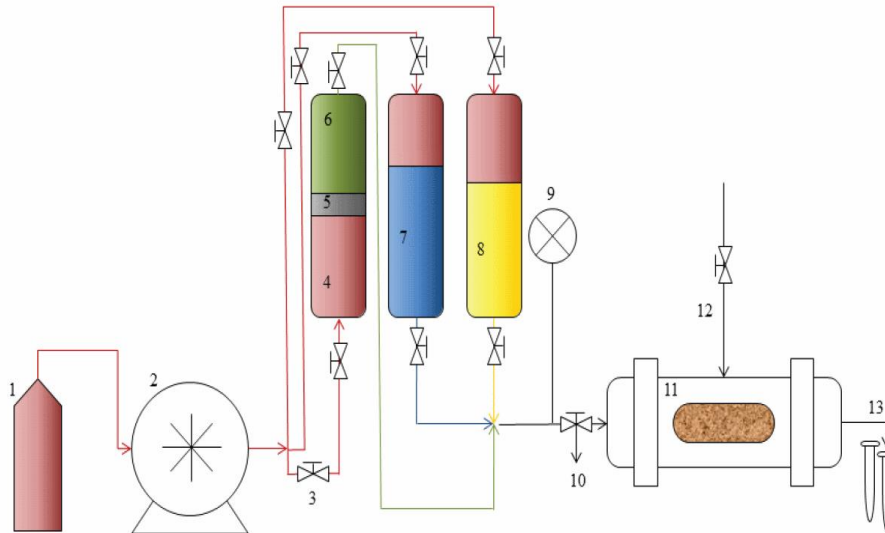

Figure 1: Experimental setup of the core flooding apparatus [7]

2.6.1. Data collection using core flooding equipment

In carrying out this experiment, the actual reservoir system has to be reproduced at the laboratory scale. The core plugs used for this experiment were already saturated 100% with brine after the cleaning procedure; therefore, there was the need to introduce oil into the core plugs. At a flow rate of 3cc/min, oil was injected into the core plugs draining out the initial brine in the core plug until no more brine was produced. This process enabled the establishment of the initial water saturation, S_{wi}. After injecting oil into the core plugs and establishing an initial water saturation value, at a rate of 3 cc/min water was injected into the core plugs. This was done in the representation of the secondary recovery process.

However, nanocomposite flooding was conducted after water flooding, considering the concentration of the recovery agents, and each value was collected every 4 minutes and recorded. To measure the oil recovery, the experiment was monitored closely all through the duration of each flooding and recovery was manually recorded to determine the amount of crude oil produced at various time intervals.

2.6.2. Nanocomposite flooding analysis

All individual core plugs were flooded using the same method. Core plugs were first injected with oil at a flow rate of 3 cc/min, displacing the brine already 100 % saturated in the core plug. This drainage process was done to attain initial water saturation and oil saturation values. This was followed by the injection of water at 3 cc/min to displace the oil in the core plug until no more oil can be produced via the water being injected. The back pressure set at 200 psi and the confining pressure having built to 700 psi at the end of the flooding.

The production of oil from the core plug into the receiving tube was monitored by observing the level of the oil-brine interface. Movement of the interface level indicated the continuous production of fluid. When oil production stopped, it implied that the core content has reached its residual oil saturation. The volume of the produced oil was recorded in cc. This concluded the secondary recovery process for the core plug, thereby making it viable for tertiary recovery.

After water flooding, the core plug was injected with nanocomposite at different concentrations, and the flooding continued until no more oil was produced from the core plug. This was done to investigate the capacity of the nanocomposite to recover the residual oil left in the reservoir after water flooding. The flooding process recovered oil continuously till only brine was being produced.

3. Results and discussion

3.1. Viscosity profile of nanocomposites

3.1.1. Effect of aluminium oxide nanoparticle on the viscosity of xanthan gum

The effect of aluminium oxide nanoparticle on 0.1, 0.5 and 1.0 wt% of xanthan gum at 10.21 s^{-1} shear rate were investigated and presented in the plot (Figure 2).
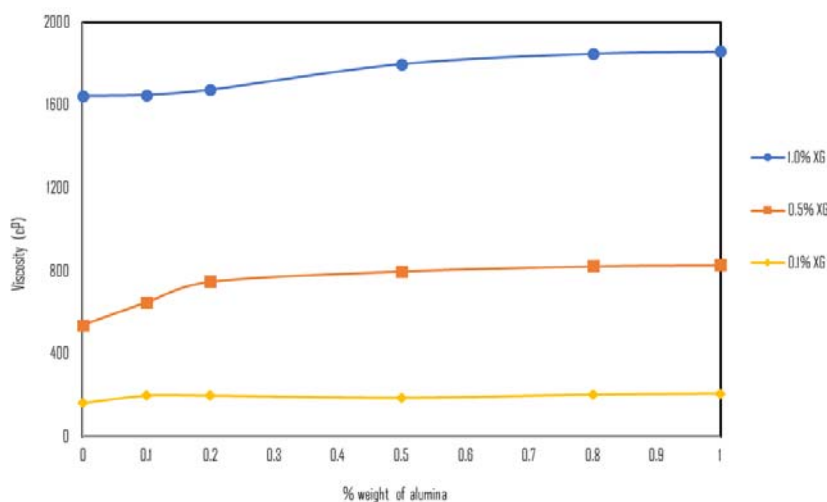

Figure 2: Viscosity profile of alumina-xanthan gum nanocomposite

3.1.2. Effect of aluminium oxide nanoparticle on the viscosity of guar gum

The effect of aluminium oxide nanoparticle on 0.1, 0.5 and 1.0 wt% of guar gum at 10.21 s^{-1} shear rate were investigated and presented in the plot (Figure 3).
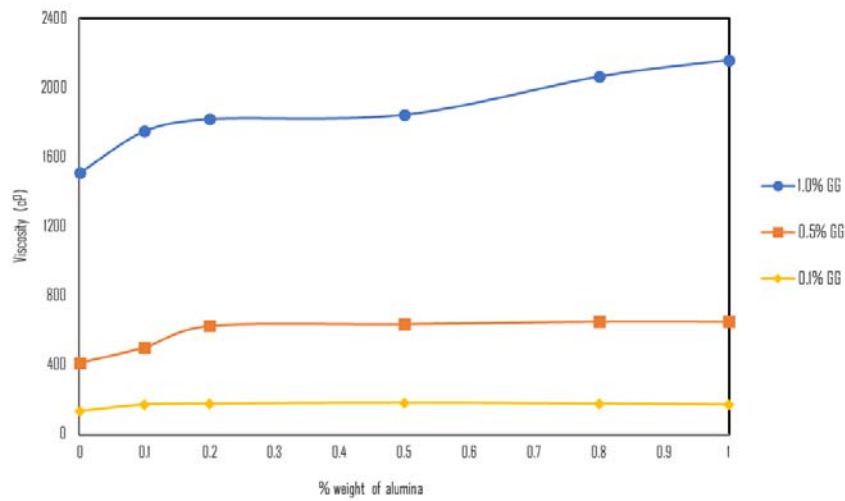
Figure 3: Viscosity profile of alumina-guar gum nanocomposite

3.2. Crude oil recovery potential of nanocomposites

3.2.1 Nanocomposite flooding for alumina and xanthan gum polymer

The core plug was first flooded with water and then flooded with a nanocomposite of aluminium oxide nanoparticle and xanthan gum polymer at 28°C. Xanthan gum at 0.5, 1.5, and 2.5 g was dissolved in 500 mL distilled water indicated as 0.1, 0.3, and 0.5 wt%. Each of these was mixed with three concentrations of aluminium oxide and used for flooding. The results are presented in figures 4 to 6.
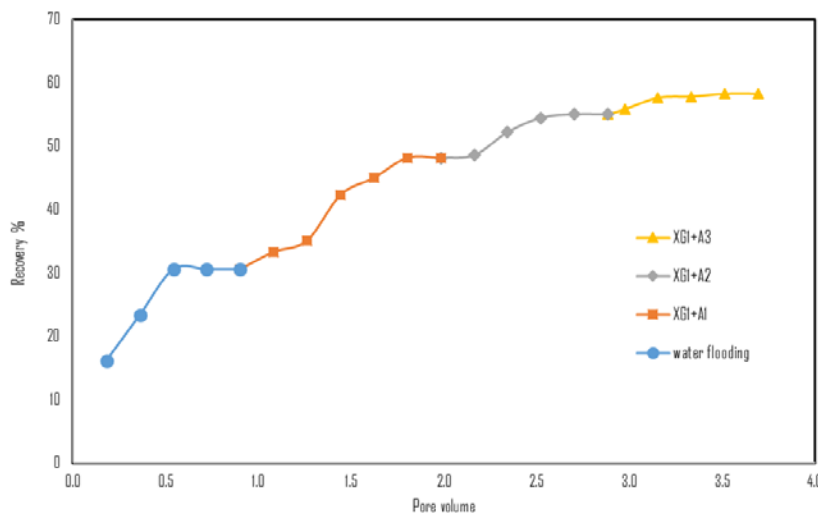

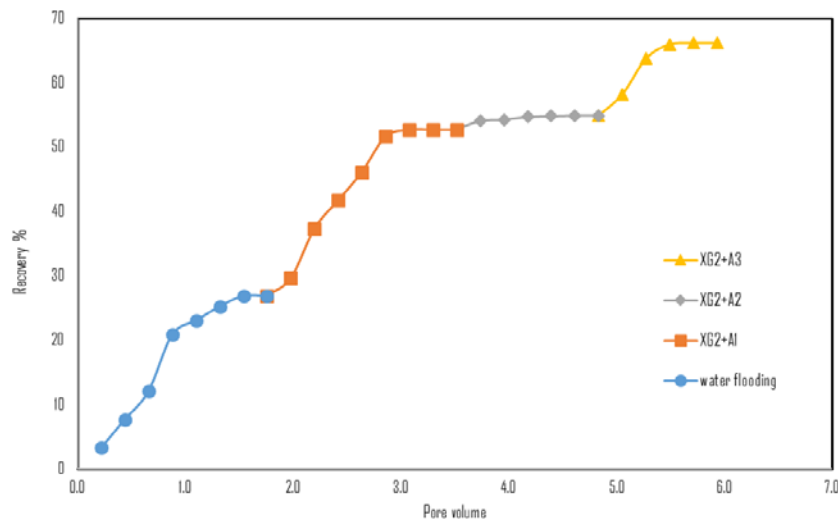**Figure 4:** Nanocomposite flooding for alumina and 0.1 wt% xanthan gum

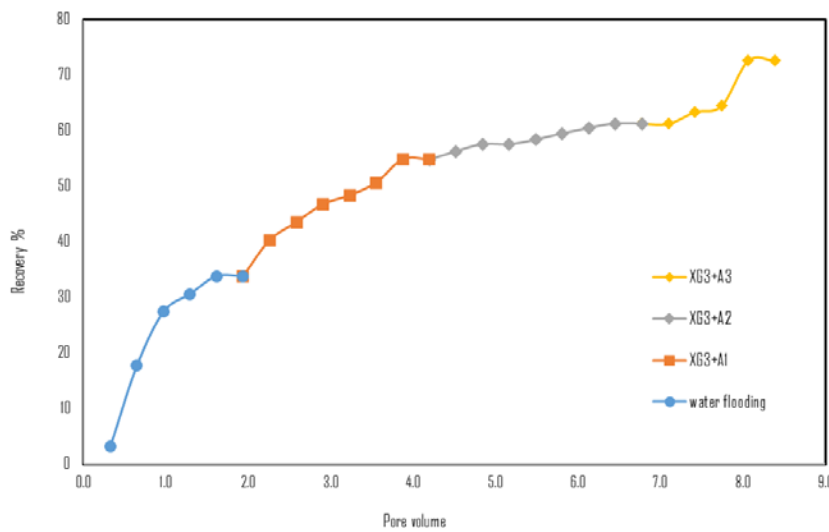**Figure 5:** Nanocomposite flooding for alumina and 0.3 wt% xanthan gum

Figure 6: Nanocomposite flooding for alumina and 0.5 wt% xanthan gum

3.2.2. Nanocomposite flooding for alumina and guar gum polymer.

The core plug was first flooded with water and then flooded with a nanocomposite of aluminium oxide nanoparticle and guar gum polymer at 28°C. Guar gum at 0.5, 1.5, and 2.5 g was dissolved in 500 mL distilled water indicated as 0.1, 0.3, and 0.5 wt%. To carry out the flooding, the lowest concentration of polymer was first mixed with different weight increments of aluminium oxide nanoparticle i.e. 0.1, 0.3, and 0.5 wt%. The flooding result illustrates the effect of the nanocomposite on various concentrations of the polymer. The results are presented in figures 7 to 9.
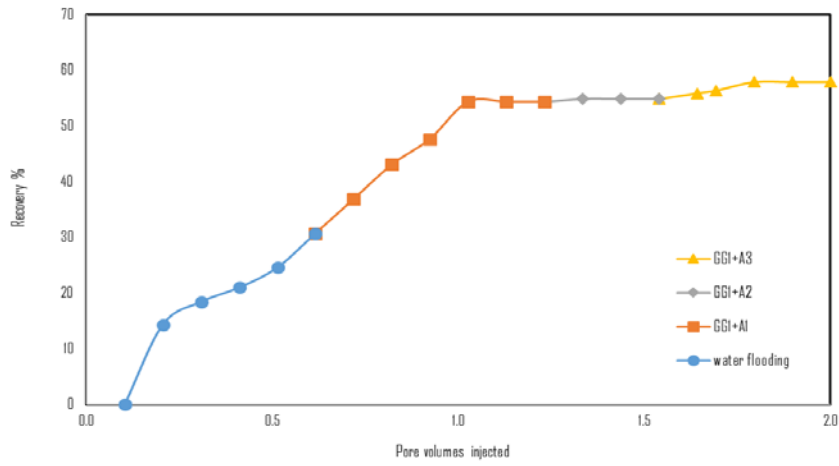
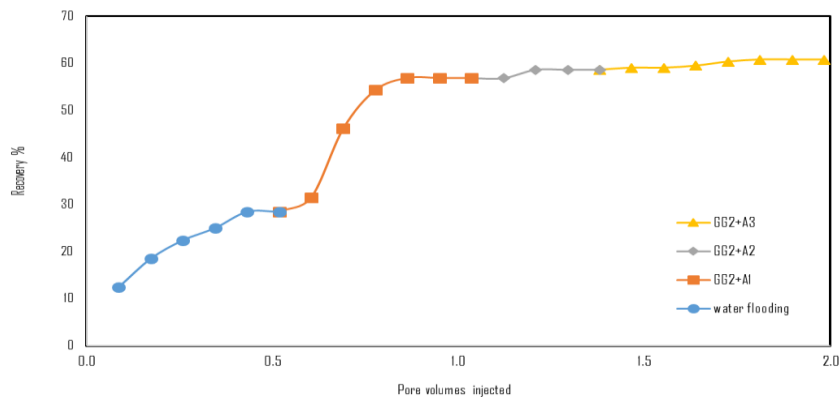**Figure 7:** Nanocomposite flooding for alumina and 0.1 wt% guar gum

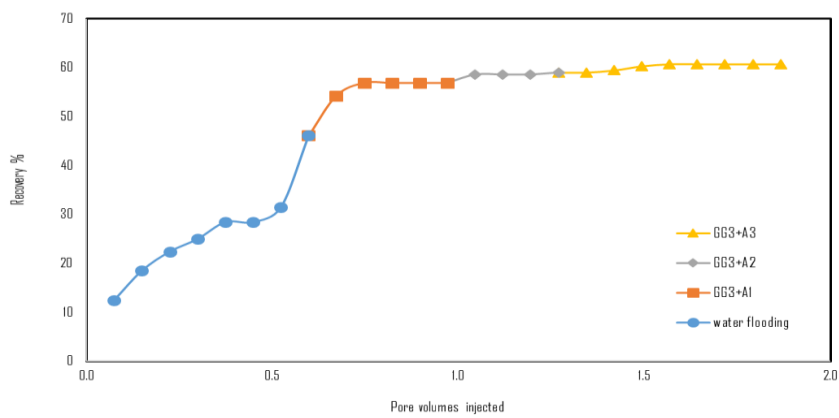**Figure 8:** Nanocomposite flooding for alumina and 0.3 wt% guar gum

Figure 9: Nanocomposite flooding for alumina and 0.5 wt% guar gum

3.3. Discussion

3.3.1. Result for viscosity tests of the nanocomposites

Viscosity was measured for two nanocomposites: alumina-xanthan gum and alumina-guar gum. The weight percent of alumina was varied up to 1.0 for different concentrations of the polymer. For both nanocomposites, it was observed that the viscosity of the polymers increased with increasing concentration of aluminium oxide nanoparticle. This implies that aluminium oxide nanoparticle increases the bonds of polymers creating an increase in intermolecular attraction. For both polymers, aluminium oxide nanoparticle is seen to have the potential to increase its viscosity by around 50 %, even though the nanoparticle has a slightly higher effect on guar gum.

3.3.2. Result analysis for alumina-xanthan gum polymer flooding

The effect of aluminium oxide nanoparticle on the oil recovery potential of xanthan gum was investigated at three different concentrations of xanthan gum and three concentrations of the nanoparticle. Water flooding was first carried out, followed by sequential flooding with three concentrations of aluminium oxide. The concentrations used are shown in table 1.

From figure 4, we observe that water flooding achieved 30.6 % recovery leaving a significant amount of oil in the core plug. The first concentration of xanthan gum was then flooded with three concentrations of alumina. the first concentration of alumina achieved 48.2 % recovery and then no more oil was recovered. It was then flooded with a higher concentration of alumina which achieved 55 % total recovery, while the highest percentage achieved 58.5 % total recovery.

A higher percentage of xanthan gum was then used, and the result is shown in figure 5. The mixture with the lowest concentration of alumina achieved recovery of 52.7 % after recovery. The next concentration achieved recovery of 54.9 % while the highest concentration of alumina achieved 66.2 %. Figure 6 shows the flooding result obtained for alumina and the highest percentage of xanthan gum. The three concentrations of alumina achieved 54.8, 61.3 and 72.6 % total recovery respectively.

The results indicate that oil recovery increased with the concentration of xanthan gum polymer. This is an indication of higher sweep efficiency and lower viscous fingering as the concentration of xanthan gum increases. In a similar vein, total oil recovery was observed to increase with the concentration of aluminium oxide nanoparticle. This indicates that aluminium oxide nanoparticle has the potential to increase oil recovery capacity of xanthan gum.

3.3.3. Result analysis for alumina-guar gum nanocomposite flooding

To evaluate the effect of aluminium oxide nanoparticle comparatively, it was flooded in combination with guar gum. Similar to xanthan gum, three concentrations of guar gum were mixed with three concentrations of xanthan gum and used for flooding. The results are shown in figures 7-9 and the concentrations are shown in table 1.

In figure 7, the lowest concentration of guar gum, GG1 was flooded with three concentrations of aluminium oxide after water flooding and this achieved a total crude oil recovery of 54.4, 54.9 and 57.9 % respectively. For GG2, the different concentrations of aluminium oxide achieved total crude oil recovery of 56.9, 58.6 and 60.8 % respectively. For GG3, the different concentrations of aluminium oxide achieved total crude oil recovery of 59, 66.4 and 69.4 % respectively.

In comparison to the xanthan gum-alumina nanocomposite, it was observed that crude oil recovery increased with the concentration of guar gum polymer; however, total recovery achieved from xanthan gum

was higher than that obtained from guar gum polymer, indicating that xanthan gum has higher volumetric efficiency and experiences less viscous fingering than guar gum. Also, the total recovery of guar gum increased with increasing concentration of aluminium oxide nanoparticle; but the nanocomposite of xanthan gum and aluminium oxide recorded higher recoveries than that of guar gum and aluminium oxide.

4. Conclusion

Aluminium oxide nanoparticle worked effectively as a good EOR agent when combined with xanthan gum and guar gum. It was found to increase the viscosity of both polymers and increase the oil recovery produced using the two polymers. Several experimental runs were performed to examine the activity of each solution. A viscosity device and core flooding equipment were used to obtain results adopted in this article to investigate the EOR mechanisms and viscosity of each solution. The following conclusions are obtained from the research:

1. Aluminium oxide nanoparticle increased the viscosity of xanthan gum and guar gum polymers by about 50 %, with xanthan gum having a higher viscosity than guar gum
2. Xanthan gum has higher crude oil recovery than guar gum with both experiencing higher recoveries as concentration was increased.
3. At 28°C, xanthan gum and aluminium oxide mixture achieved the highest total recovery of 72.6 % while the guar gum and aluminium oxide mixture achieved the highest recovery of 69.4 %.

Xanthan gum and guar gum have a high potential for crude oil recovery, and both polymers become more effective with the addition of aluminium oxide nanoparticles. Aluminium oxide nanoparticles can dissipate heat effectively when in fluids, and has high resistance against temperature and shear forces. It also can increase the viscosity of polymer fluids and can neutralize cations in brine, giving it resistance to high salinity conditions. Aluminium oxide nanoparticle is therefore considered a viable option for crude oil recovery using nanocomposites.

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