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Physicochemical Characterization of Zirconia Nanoparticle-Based Sodium Alginate Polymer Suspension for Enhanced Oil Recovery

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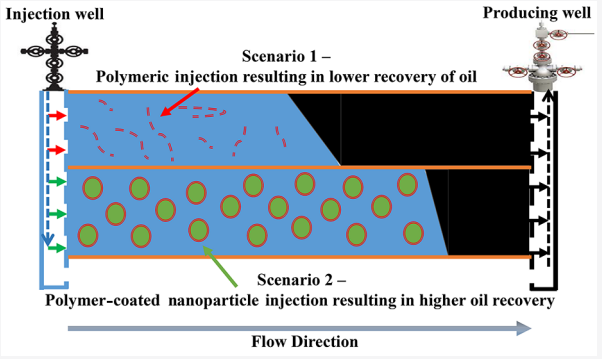
ABSTRACT: Biopolymers have been employed in enhanced oil recovery (EOR) due to their high viscosity and significant effects on waterflooding performance. Sodium alginate (NaAlg) is an excellent biopolymer that is extracted primarily from brown algae. It has been used in the biotechnology industry as a thickening agent, colloidal stabilizer, and oil recovery application. In the present study, a series of sodium alginate/zirconium oxide nanoparticle suspensions were prepared via solution mixing, and the effect of nanoparticle content, polymer concentration, temperature, salinity was investigated on the rheological behavior using a concentric cylinder dynamic rheometer. The rheology results revealed that the lower concentration of sodium alginates (0.01 to 0.06 wt %) and sodium alginate/nanoparticle suspensions (0.02 to 0.06 wt %) displayed shear thinning behavior for the whole range of shear rate from 0.1 to 100 s^{-1}. It was noticed that the aging of the polymer/nanoparticle suspension at 25 °C for 7 days did not significantly affect the rheological characteristics. It was moreover observed that enhancing salinity from 0.015 to 0.1 wt % in the 0.04 wt % ZrO_2 nanoparticle suspension comprising 0.1 wt % sodium alginate showed a progressive decrease in viscosity under the temperature range from 25 to 50 °C. The proposed sodium alginate/zirconium oxide nanoparticles from the mentioned results indicate their potential for enhanced oil recovery applications.



1. INTRODUCTION

The hydrocarbon industry is focusing on new ways for energy storage and its production;[1−6] among the various energy sources, crude oil remains the predominant resource presently.[7−9] Furthermore, demand for crude oil has been rising worldwide; while discovering new oil reservoirs is quite difficult, 30%−60% of oil remains trapped in the currently producing reservoirs.[10,11] Hence, researchers have focused on extracting the remaining oil in the matured reservoirs using additional technologies, such as nanotechnology.[12−15]

Nanotechnology is considered a complete game changer in exploiting oil and gas resources and will significantly contribute to fossil-based energy technologies developed over the next 30 years.[16] Among the technological advancements in nanotechnology, a new type of fluids called "smart fluids" or nanofluids have recently become more predominant in the oil and gas industry.[17,18] Nanofluids are nanoparticles (NPs) suspended in a base fluid.[19,20] The nanofluids are formulated by nanoparticle addition to base fluids for intensification and improvement of rock surface properties. Significantly, nanofluid properties depend on the surface properties of the nanoparticles. Since suspensions of nanodimensional particles exhibit greater sedimentation stability, nanofluids' properties are better than the properties of conventional fluids.[21]

Enhanced oil recovery (EOR) is usually performed to minimize the remaining residual oil saturation and improve oil recovery and thus improve reserves.[22−27] Polymer flooding is one of the most efficient chemical methods to produce heavy oil.[28,29] In polymer flooding, slugs of viscous polymer formulation are injected to displace the oil toward the production well. This method optimizes the mobility ratio, which enhances vertical and areal sweep efficiencies due to inhibition of the viscous fingering phenomenon.[30−33] Thus, polymer flooding is the main focus of the present investigation.[34−39]

We further conducted a comparative property analysis of our proposed materials (nanoparticle, additive/polymer, and

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Table 1. Comparison of Presented Materials for EOR

Study no.	Study	Nanoparticle	Additive	Salinity (ppm)
1	Effect of nano titanium dioxide on heavy oil recovery during polymer flooding[40]	Titania	Hydrolyzed polyacrylamide	20,000
2	Enhancing rheological properties of hydrophobically associative polyacrylamide aqueous solutions by hybriding with silica nanoparticles[30]	Silica	Hydrolyzed polyacrylamide	32,868
3	Appraising the impact of metal-oxide nanoparticles on rheological properties of HPAM in different electrolyte solutions for enhanced oil recovery[41]	Alumina, Silica, and Titania	Hydrolyzed polyacrylamide	10,000−60,000
4	Synergistic effect of nanoparticles and polymers on the rheological properties of injection fluids: implications for enhanced oil recovery[20]	Zirconia	Polyacrylamide	0−10,000
5	Controlled foaming of polymer films through restricted surface diffusion and the addition of nanosilica particles or CO_2-philic surfactants[42]	Silica	Poly(methyl methacrylate)	0
6	Interfacial dynamics and rheology of polymer-grafted nanoparticles at air−water and xylene−water interfaces[43]	Silica	Poly(2-(dimethylamino) ethyl methacrylate)	584
7	Rheology and structure formation in diluted mixed particle- surfactant systems[44]	Silica	Cetrimonium bromide	0
8	Rheology and polymer flooding characteristics of partially hydrolysed polyacrylamide for enhanced heavy oil recovery[45]	N/A	Hydrolyzed polyacrylamide	0−50,000
9	Novel smart water-based titania nanofluid for enhanced oil recovery[46]	Titania	Polyethylene glycol and polyvinylpyrrolidone	40,572−198,429
10	Physicochemical characterization of zirconia nanoparticle-based sodium alginate polymer suspension for enhanced oil recovery (present study)	Zirconia	Sodium alginate	500−15,000

salinity) with literature proposed for potential application of EOR (Table 1).

The in situ tests for performance analysis of formulations were tested with two standard equipments (rheometer and zeta analyzer).[20,47,48] Thus, new materials were tested in this work, and their potential applications for EOR are discussed in this work.

Polymer−silica nanoparticle composites are thermally stable, water soluble, and better transportable in a porous medium.[49] Synthesis of SiO_2 and a water-soluble ethylene oxide-based nanocomposite with enhanced viscosity has been reported.[50] Nonaqueous/nanofluid-polymer formulations can also be used as fracturing fluids to unlock tight gas or shale gas.[51−56] Researchers have consistently studied the effect of different NPs on the rheological characteristics of nanofluids (NFs) for EOR purposes. The effect of various nanoparticles such as SiO_2, Al_2O_3, and TiO_2 containing NFs on the viscosity behavior of oil has been investigated.[57] They observed a change in the viscosity of oils after each nanoflooding experiment. Yang et al. synthesized oleic acid modified titania (OA-TiO_2) nanoparticles for enhanced oil recovery in low permeability reservoirs.[58] These results revealed that oleic acid-modified titania nanoparticles exhibited great potential in reducing injection pressure and facilitating hydrocarbon recovery in low-permeability reservoirs. Furthermore, Al_2O_3-containing NF showed the highest viscosity reduction, and SiO_2 exhibited a lower change in viscosity reduction at all investigated temperatures ranging from 26 to 60 °C.[59] Nevertheless, the literature on the role of ZrO_2 NPs in EOR applications is limited.

The oil and gas industry has actively sought a polymer that can tolerate high salinity and high temperature. Most of the commonly used polymers for EOR are synthetic polymers such as polyacrylamide and biopolymers such as xanthan gum, starch, scleroglucan, hydroxyethyl cellulose (HEC), carboxymethyl cellulose (CMC), guar gum, cellulose, and lignin.[60−62] In the past few years, biopolymers have attracted the attention of the petroleum industry.[36,63,64] It has also been reported that biopolymers have potential applications in EOR due to their high viscosity and effect on the water-flooding performance in EOR.[65,66] Biopolymers such as xanthan gum have been reported to have excellent performances under high salinity brine conditions and are highly compatible with injected fluid additives for EOR applications.[67] Olabode et al. formulated a biopolymer from *Solanum tubersome* waste starch for oil recovery applications which served as a substitute for chemically synthesized polymers.[68] The polymer showed high reservoir temperature, mobility, and excellent rheological properties. Orodu et al. used biopolymers potato starch and gum arabic along with Al_2O_3 nanoparticles for enhanced oil recovery applications.[69] The biopolymer nanocomposites displayed improved viscosity over the biopolymers. The authors found that a potato peel starch nanocomposite (PSPNP) exhibited superior viscosifying efficiency and strong shear thinning behavior compared to another biopolymer. Rellegadla et al. employed biopolymer xanthan gum with nickel nanoparticles for enhanced oil recovery applications.[70] Xanthan-nickel nanoparticles possessed a higher intrinsic viscosity of 55.25 dL/g than the viscosity of 49.13 dL/g for the gum solution. Their results revealed that the novel nanoparticle assisted the polymer flooding method and enhanced oil recovery. Recently biopolymers have been explored for oil-contaminated soil remediation instead of biosurfactants.[71] Nevertheless, studies on the synergistic effect of biopolymer sodium alginate and nanoparticles on rheological behavior are limited.

In order to produce incremental oil recovery, it is essential to identify new EOR agents that are efficient, economical, and environmentally friendly. Therefore, the present study investigates the effect of a new biopolymer, sodium alginate and ZrO_2 nanoparticles, on various factors such as salinity, temperature, concentration, and rheological characteristics of a variety of polymer nanofluids to investigate the potential of polymer nanofluids as EOR agents.

2. METHODS AND MATERIALS

Rheological experiments were performed in a combined motor and transducer rheometer (CMT rheometer; model: discovery hybrid rheometer series DHR-3). The geometry of the rheometer consisted of a DIN concentric cylinder of Peltier steel (bob diameter and length of 28 and 42 mm, respectively). The zeta potential measurements were conducted with a zeta nano sizer (ZN3600, Malvern Instruments).[72] All measurements were repeated thrice to assess the uncertainty associated with the experiments.[73]

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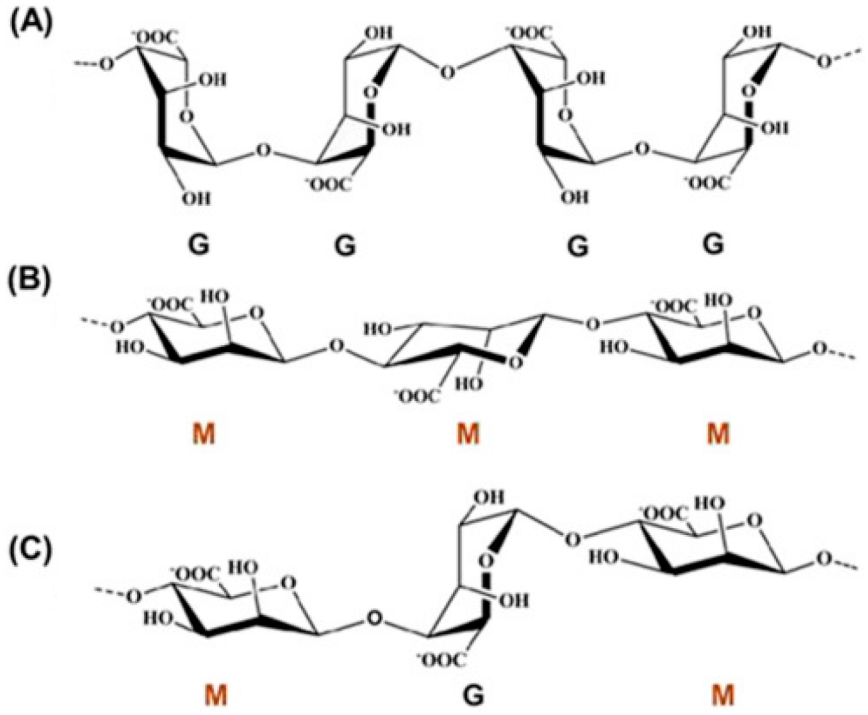
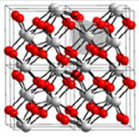


Figure 1. Sequences of alginates: (a) homopolymeric blocks of poly-α-1,4-l-guluronic acid (GG), (b) homopolymeric blocks of poly-β-1,4-d-mannuronic acid (MM), (c) heteropolymeric blocks of alternating M and G residues. Reprinted from ref 79. Copyright 2021, with permission from Elsevier.

Table 2. Characteristics of Zirconium Dioxide Nanoparticles

Chemical Formula	Primary particle size (nm)	Chemical Structure Depiction	Specific surface area ($m^2.g^{-1}$)	Purity (wt.%)
ZrO_2	<100		≥ 25	99.9 %
Density (g/cc)	**Molecular mass (g/mol) @ 298.15 K**	**Boiling point (K)**	**Melting point (K)**	**Appearance**
5.89	123.22	5273.15	2973.15	White powder

Several chemicals (all analytical grade) were used in the experiments, as follows:

(1) Sodium alginate ($C_6H_9NaO_7$; represented as NaAlg; Sigma-Aldrich) is of purity 99.9 wt %. Sodium alginate is a sodium salt form of alginic acid and gum, used as a biopolymer extracted primarily from brown algae.[74] Commercially available sodium alginate is usually extracted from various seaweeds.[75] The chemical composition and sequence distribution of sodium alginate depend on the species and parts of the seaweed employed for extraction. It is a linear, unbranched, amorphous copolymer composed of β-D-mannuronic acid (M) and α-L-guluronic acid (G) linked by 1→4 glycosidic bonds. The M and G units in the alginates may be randomly or nonrandomly organized as heterogeneous or homogeneous sequences;[76] alginate sequences can be seen in Figure 1. Sodium alginate polymer has been miscible with other polymers and has excellent suspending properties; hence, it could be an ideal candidate for application to EOR projects.[77,78]

(2) Sodium chloride (purity ≥99.5 mol %; NaCl; Rowe Scientific) and zirconium oxide (nanoparticles; insoluble in water; ZrO_2; procured from Sigma-Aldrich) were used. The detailed properties of the nanoparticles can be found in Table 2.

(3) Note that for all aging experiments, measurements were conducted with 0.05 wt % NaCl brine for 7 days to assess the effect of aging on the rheology of the polymer solutions. Additionally, we studied the effect of salinity on the rheological behavior of dispersions. Four suspensions (0.05, 0.5, 1, and 1.5 NaCl wt %) were tested with a constant concentration of 0.06 wt % sodium alginate.

(4) Sodium hydroxide (100 wt % purity, NaOH, Rowe Scientific) was used to buffer the pH of the solutions. The base fluid was DI water (Merch Millipore, resistivity 18.2 MΩ cm).

The samples were aged in closed-lid beakers to avoid cross-contamination and were tested for rheological properties, all at 25 °C and atmospheric pressure (see Table 3 for a detailed test matrix).

The solutions were buffered (using 0.01 M NaOH droplets as required) to the desired pH of 8 ± 0.2 and used in all experiments as sodium alginate polymer remains stable at pH 5−11.[80] Suspensions

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Table 3. Test Design Matrix

Study no. and study (figure no.)	Independent variable	Dependent variable	Formulations (wt %) NaAlg	NaCl	ZrO_2	pH
1. Effect of sodium alginate concentration and aging (Figure 2)	Shear rate (10−100 s^{-1})	Apparent viscosity (Pa s)	0.01−0.2	0.05	0	8
2. Effect of salinity and temperature (Figure 3)	Shear rate (10−100 s^{-1})	Apparent viscosity (Pa s); ramp up and down	0.06	0.05−1.5	0	8
	Temperature (25−45 °C)	Apparent viscosity (Pa s)	0.1	0.05−0.1	0	8
3. Stability of zirconia nanofluids (Figure 4)	pH (6−12 pH)	Zeta potential (mV)	0	0.05	0.01−0.2	6−12
4. Effect of salinity, sodium alginate, and zirconia NP (Figure 5)	Temperature (25−55 °C)	Apparent viscosity (Pa s)	0	0.5	0.05−0.5	8
			0.06	0.05	0.08−0.5	8
			0.1	0.5	0.05−0.5	8
			0.1	0.05	0.2−0.5	8

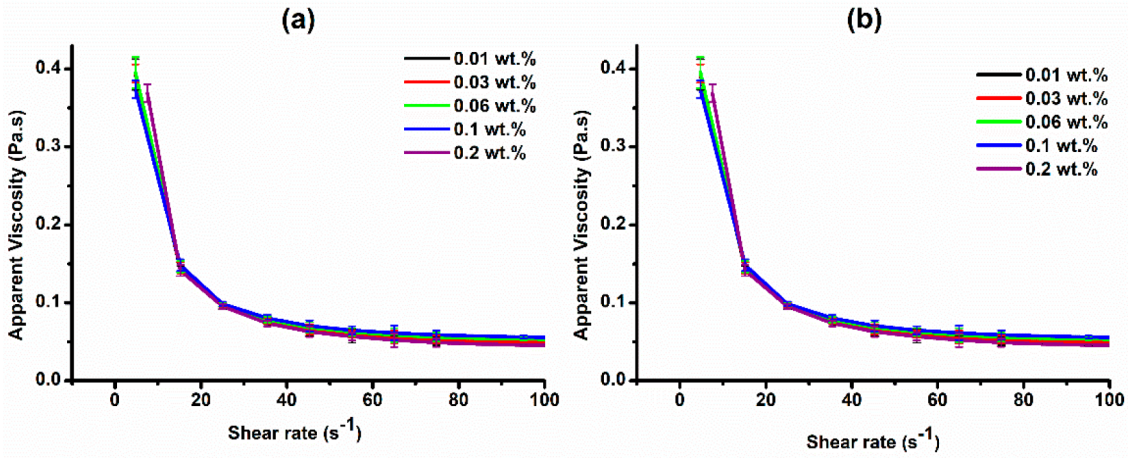

Figure 2. Apparent viscosity as a function of shear rate at various sodium alginate concentrations (0.01−0.2 wt %): (a) before aging and (b) after aging in sodium alginate for 7 days.

were formulated with DI water-based fluid and were mixed with the polar additives (sodium chloride and sodium alginate) and a nonpolar additive (zirconia NPs) to formulate a dispersed suspension.[81] For all NPs loadings, sonication continued for 10 min at 200 W to ensure efficient dispersal of the solid NPs in the base liquid. Note that the sonication time was opted due to the solutions' colloidal nature. The probe's initial temperature was set at 24 °C, and the final temperature noted was 26 °C, which was verified using an infrared laser thermometer. In order to minimize the contamination of the ultrasonic homogenizer titanium tip, an abrupt increase in temperature pulse (i.e., interruption in ultrasonication) after every 10 s for 5 s was activated. Further, the titanium tip was cleaned with ethanol and water for each newer suspension.

3. RESULTS AND DISCUSSION

As described in Section 2, four types of analyses were conducted on various formulations: (i) sodium alginate concentration (0.01−0.2 wt %) and aging (7 days), (ii) salinity (0.015−1.5 wt % NaCl), (iii) stability of nanoformulation (0.01−0.2 wt %), and (iv) various ZrO_2 nanofluid−polymer formulations; (a) no polymer, 0.5 wt % NaCl, (b) 0.06 wt % NaAlg, 0.05 wt % NaCl, (c) 0.1 wt % NaAlg, 0.05 wt % NaCl, and (d) 0.1 wt % NaAlg, 0.5 wt % NaCl (Table 3). The results were explained scientifically and supported by the literature data, where applicable.

3.1. Effect of Sodium Alginate Concentration. Various sodium alginate concentrations (0.01−0.2 wt %) were tested (at 0.05 wt % NaCl concentration, 25 °C, atmospheric pressure, shear rates from 0.1 to 100 s^{-1}, and pH value 8). No viscosity/shear rate changes were observed when the sodium alginate (NaAlg) concentration increased from 0.01 to 0.2 wt % (Figure 2a). However, a drastic decrease in apparent viscosity with an increase in shear rate was observed. This shear-thinning behavior is consistent with literature data which showed a similar trend for polyacrylamide and sodium alginate/guar gum mixtures.[77,82]

However, a further increase in shear rate from 20 to 100 s^{-1} resulted in an insignificant change for all sodium alginate concentrations (0.01−0.2 wt %). Shear thinning has enormous significance in EOR applications; thus, the shear rate in a large pore throat is lower than in a small pore throat. Hence, shear thinning leads to smaller resistance in low permeable cores and contributes to greater oil recovery in the heterogeneous reservoirs.

Furthermore, it revealed that aging for 7 days at 25 °C had no significant effect on the viscosity, Figure 2b.

At a shear rate greater than 40 s^{-1}, the apparent viscosity lowers, which is advantageous from an injection standpoint; however, at lower shear rates (<40 s^{-1}), the viscosity is higher, which aids in increased mobility ratio and yields a higher recovery factor. Mechanistically, polymer solution shear thinning is induced by the disentanglement of polymer chains when the shear rate is increased.[83] Polymers with high molecular weight are orientated randomly and entangled. These highly anisotropic polymer chains start to disentangle and align along the shear direction when sheared at a higher rate (in our case >40 s^{-1}).[84] As a result, there is less molecular/

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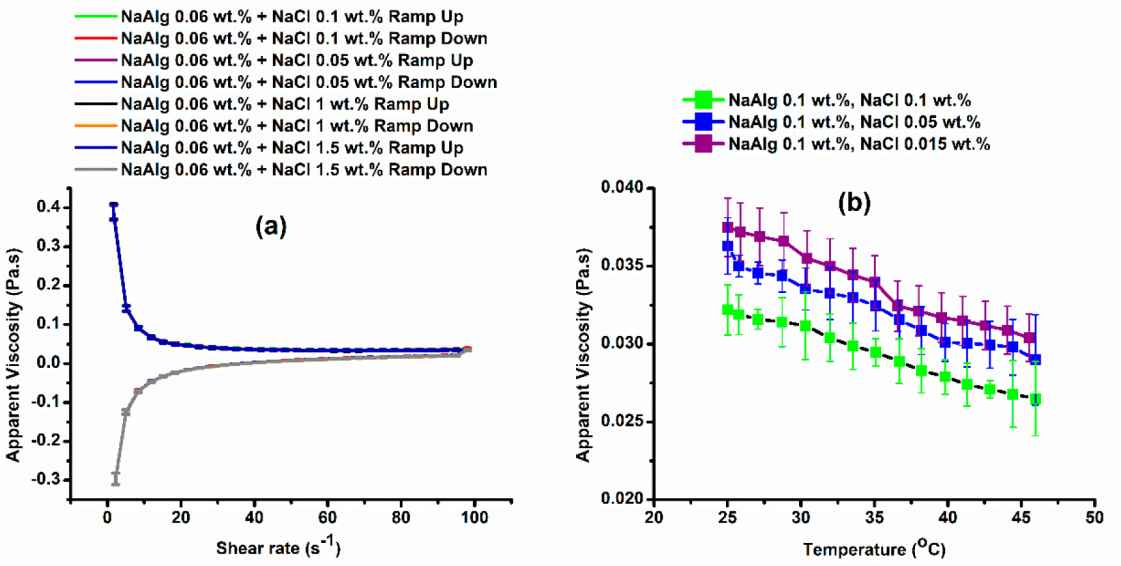

Figure 3. Apparent viscosity as a function of (a) shear rate with 0.06 wt % sodium alginate and various salinities from 0.05 to 1.5 wt % NaCl and (b) temperature at pH 8 with 0.1 wt % sodium alginate and salinities from 0.05 to 0.1 wt % NaCl.

particle contact and more free space, lowering viscosity.[85] Typically, polymer solutions employed in EOR processes are shear-thinning fluids whose apparent viscosities decrease in a reservoir with increasing shear rate. This was beneficial from a polymer/nanofluid injectivity standpoint since the viscosity near the injection well was lower, which provided more favorable productivity and recovery of hydrocarbons. Once the polymer moved far into the reservoir, shear rates declined, and the viscosity increased, which provided the desired mobility control desired.

3.2. Effect of Salinity. The effect of salinity (0.1−1.5 wt %) with the shear rate for a 0.06 wt % sodium alginate polymer was examined. Additionally, 0.1 wt % sodium alginate was tested for its apparent viscosity response from 25 to 45 °C with 0.015−0.1 wt % low-salinity loading.

The viscosity declined exponentially when the shear rate was increased from 0.1 to 100 s^{-1}. Further, a change in salinity from 0.05 to 1.5 wt % did not affect the curves representing apparent viscosity (Figure 3a). Furthermore, the viscosity decreased linearly with an increase in temperature from 25 to 47 °C (Figure 3b). The system's viscosity for 0.1 wt % NaCl solution was lowest throughout the tested temperature range compared to 0.05 and 0.015 wt %. Figure 3b also revealed that the apparent viscosity undergoes an increase when the NaCl concentration increases from 0.015 to 0.1 wt % more strongly in the temperature range of 25 to 35 °C.

The decrease in apparent viscosity with increased salinity can be attributed to the interaction/attachment of counterions from salt (NaCl) to the polar heads of the polymer (NaAlg), which reduces their overall charge and stabilizes the system.[86−88] We hypothesized that apparent viscosity decreased upon absorbance of Na^+ cations (from NaCl), and polymeric chains of NaAlg were separated, retarding the network structure formation.[89]

The decrease in viscosity with an increase in temperature is mainly due to the reduction of the attractive binding energy between polymer molecules.[90,91] This reduction in binding energy dramatically reduces the intermolecular forces between the polar groups in the polymer structure.[92] Kurkuri et al. and Cheraghian et al. had reported the rheological behavior of dispersion of sodium alginate and guar gum mixtures at different temperatures, and they noted that the viscosity of the 3% sodium alginate polymer decreased with an increase in temperature as well as with an increase in mass% of guar gum in the blend.[77,86] Furthermore, a significant decrease in viscosity with increasing temperature was also observed for all dispersions.

3.3. Effect of Zirconium Oxide Nanoparticles. In CEOR applications, polymers significantly improve sweep efficiency and mobility control.[93,94] Nevertheless, the rheological characteristics of the polymer solutions are significantly affected by the specific reservoir conditions, namely, formation temperature and shear rates.[36,95] For significant sweep efficiency, it is essential to have better stability of the nanofluid−polymer suspension in a saline environment.[96] We thus conducted a zeta potential analysis of the nanofluid formulation at a constant salinity of 0.05 wt % without NaAlg (Figure 4).

Furthermore, increasing nanoparticle concentration decreased the zeta potential and increased the stability of the

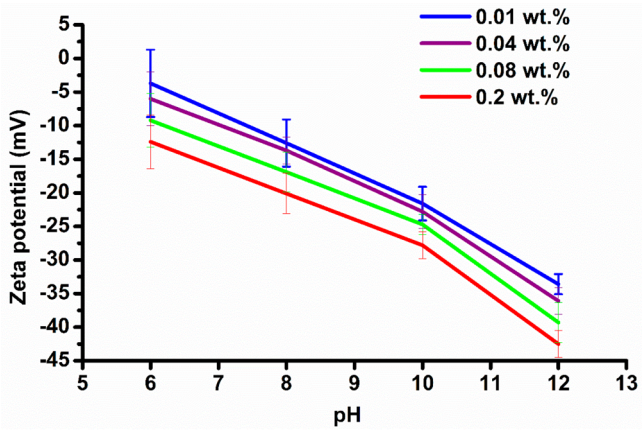

Figure 4. Zeta potential for various nanofluid formulations as a function of pH at various ZrO_2 NP loading (0.01−0.2 wt %) and 0.05 wt % NaCl, temperature = 25 °C.

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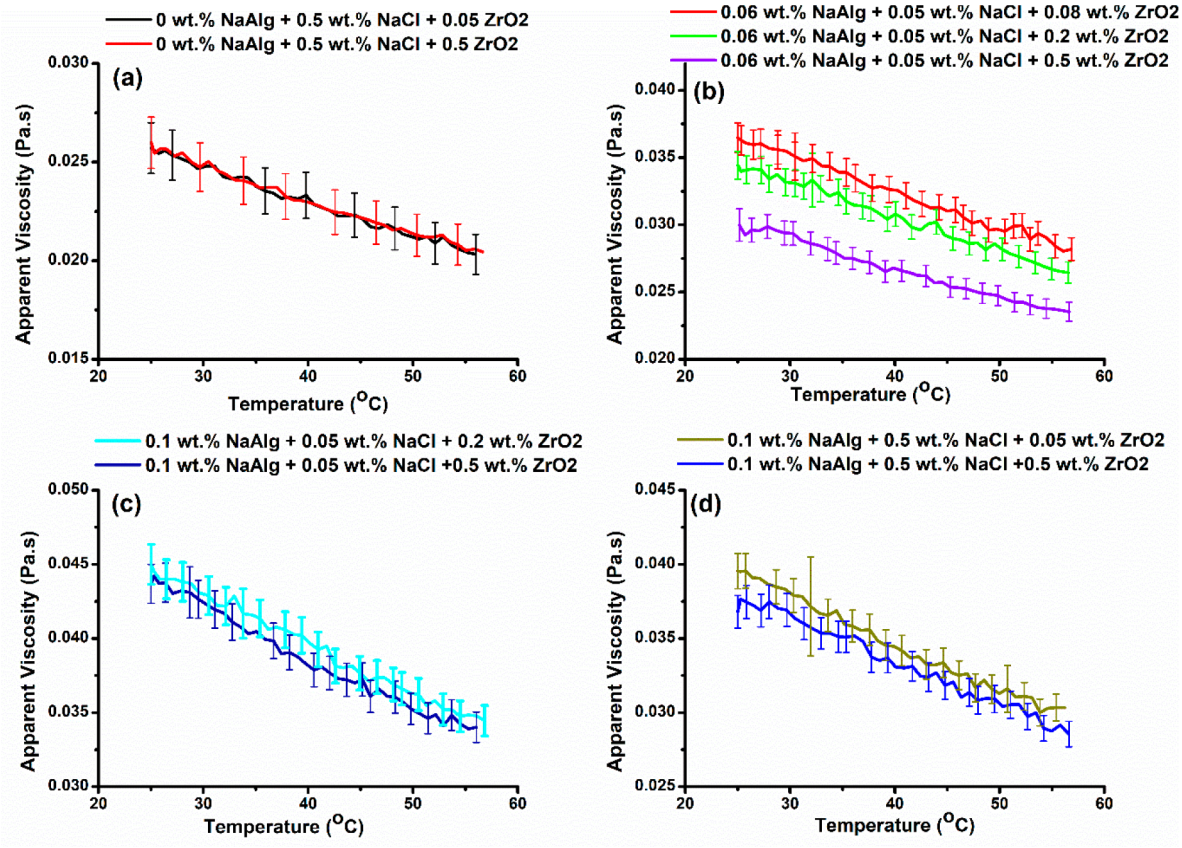

Figure 5. Apparent viscosity of various ZrO_2 nanofluid−polymer formulations: (a) no polymer, 0.5 wt % NaCl, (b) 0.06 wt % NaAlg, 0.05 wt % NaCl, (c) 0.1 wt % NaAlg, 0.05 wt % NaCl, and (d) 0.1 wt % NaAlg, 0.5 wt % NaCl.

nanoformulation (Figure 4). It has been reported by several authors that zeta potential is dependent upon nanoparticle concentration which was attributed to either a real effect or an experimental artifact.[97,98] As untreated ZrO_2 nanoparticles are usually covered with OH functional groups, the ionic state of OH^- can be affected by the pH change.[99,100] Additionally, the surface of zirconia nanoparticles adsorbed further OH^- ions as the pH is increased. Note that these further OH^- ions are from the dissociation of NaOH in the nanoformulation.

Dispersions with zeta potentials greater than +30 mV or less than −30 mV typically possess a high degree of stability.[101] At pH 8, the stability of 0.2 wt % NP nanofluid is 20 ± 3 mV, confirming that the suspension is moderately stable.[102,103] However, at pH > 10, the dispersion stability of the nanoformulation is excellent. Hence, a more stable but highly viscous polymer-coated nanoformulation is suitable for EOR applications.

Because of their better solubility and stability, stronger stabilization of foams and emulsions, and easier transport through porous surfaces, polymer-coated nanoparticles may be superior to bare nanoparticles for EOR applications.[50,62,104] Further, to assess sweep efficiency and mobility control in CEOR operations, various ZrO_2 nanoparticle concentrations (0.05−0.5 wt %) are added to the NaAlg polymer to investigate the influence of NPs on the rheological characteristics of these formulations as a function of the shear rate.[105] Figure 5a demonstrates that increasing the temperature from 25 to 55 °C results in a decrease in apparent viscosity for both concentrations of ZrO_2 (0.05 and 0.5 wt %) in the absence of polymer sodium alginate.

In general, ZrO_2 (0.08−0.5 wt %) added to the nanoformulations containing 0.015 wt % NaCl and NaAlg (0.06 wt %) caused a significant change in the apparent viscosity. The decrease in apparent viscosity in Figure 5b with increased temperature from 25 to 55 °C was more distinct and significant for the nanoformulation containing 0.5 wt % ZrO_2 than the nanoformulations containing 0.2 and 0.08 wt % ZrO_2. However, a steep decrease in apparent viscosity from 0.045 to 0.032 Pa s was observed when the temperature increased from 25 to 55 °C (Figure 5c) for both the nanoformulations containing 0.2 and 0.5 wt % ZrO_2, respectively.

An almost similar change in the apparent viscosity with temperature was noticed in Figure 5d. The figure reveals that a decrease in apparent viscosity with increasing temperature was attributed to an increase in thermal energy of the particles, which facilitated overcoming the attractive forces between them.[106] Furthermore, the decrease in viscosity can also be attributed to the enhancement in the thermal velocity of the sodium alginate molecules in the solution.[78,107]

4. CONCLUSIONS

We observed that the viscosity does not change with an increase in salinity from 0.05 to 1.5 wt % for a polymer concentration of 0.06 wt %. However, for low concentrations of NaCl (0.015−0.1 wt %), a change in viscosity was observed during the temperature ramp from 25 to 45 °C for 0.1 wt % NaAlg and 0.04 wt % ZrO_2 concentration. Further, a progressive decrease in apparent viscosity from 0.045 to 0.032 was observed with an increase in temperature from 25 to

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55 °C, and the curve remained the same for both concentrations of ZrO_2 (0.2 and 0.5 wt %).

A marked decrease in apparent viscosity with increased temperature was noticed when the polymer concentration was increased from 0.06 to 0.1 wt %. While the addition of 0.2 wt % ZrO_2 to the NaAlg concentration of 0.5 wt % shows a decrease in viscosity, indicating that the addition of nanoparticle ZrO_2 decreases the viscosity compared to the polymer suspension without NP. A steep decrease in viscosity is observed with an increase in temperature from 25 to 60 °C. In general, ZrO_2 (0.08−0.5 wt %) added to the nanoformulations containing 0.015 wt % NaCl and NaAlg (0.06 wt %) caused a significant change in the apparent viscosity. Thus, the present work demonstrates a synergistic effect between the sodium alginate polymer and zirconium oxide nanoparticles in the nanoformulation, and a higher concentration of ZrO_2 (0.5 wt %) caused a significant reduction in viscosity.

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Notes

The authors declare no competing financial interest.

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