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SDS−Aluminum Oxide Nanofluid for Enhanced Oil Recovery: IFT, Adsorption, and Oil Displacement Efficiency

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ABSTRACT: Surfactant flooding is one of the most promising chemical enhanced oil recovery (CEOR) methods to produce residual oil in reservoirs. Recently, nanoparticles (NPs) have attracted extensive attention because of their significant characteristics and capabilities to improve oil recovery. The aim of this study is to scrutinize the synergistic effect of sodium dodecyl sulfate (SDS) as an anionic surfactant and aluminum oxide (Al_2O_3) on the efficiency of surfactant flooding. Extensive series of interfacial tension and surfactant adsorption measurements were conducted at different concentrations of SDS and Al_2O_3 NPs. Furthermore, different surfactant adsorption isotherm models were fitted to the experimental data, and constants for each model were calculated. Additionally, oil displacement tests were performed at 25 °C and atmospheric pressure to indicate the suitability of SDS−Al_2O_3 for CEOR. Analysis of this study shows that the interfacial tension (IFT) reduction between aqueous phase and crude oil is enhanced considerably by 76%, and the adsorption density of SDS onto sandstone rock is decreased remarkably from 1.76 to 0.49 mg/g in the presence of these NPs. Although the effectiveness of NPs gradually increases with the increase of their concentration, there is an optimal value of Al_2O_3 NP concentration. Moreover, oil recovery was increased from 48.96 to 64.14% by adding 0.3 wt % NPs to the surfactant solution, which demonstrates the competency of SDS−Al_2O_3 nanofluids for CEOR.
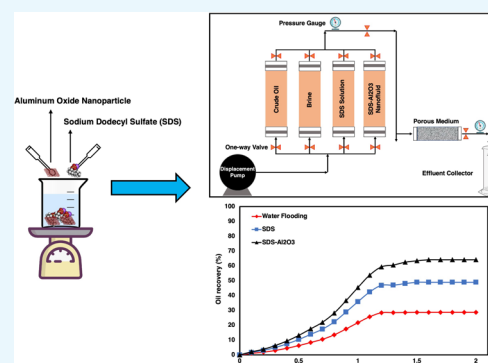
1. INTRODUCTION

Oil production mechanisms are generally divided into three phases: primary, secondary, and tertiary. Primary and secondary recovery techniques can produce only 30% of the original oil in place (OOIP).[1,2] Therefore, tertiary recovery or enhanced oil recovery (EOR) approaches are applied to recover the remaining oil in reservoirs, which cannot be performed by conventional methods. In this regard, chemical EOR (CEOR) has been considered one of the most promising EOR methods because of its higher performance and technical feasibility compared to other EOR techniques such as thermal and gas flooding.[3] Among the used chemicals in CEOR, surfactants, polymers, alkalis, and/or their synergy were found to be advantageous to produce residual oil in reservoir rocks.[2,4] The mechanism of each chemical type to enhance oil recovery is different; for example, polymers on increasing the viscosity of the displacing fluid reduce the mobility ratio,[5] and surfactant by reducing interfacial tension (IFT) and alteration wettability toward the water-wet state[6] enhance macroscopic sweep and microscopic displacement efficiency of crude oil.[7]

Surfactant flooding is reviewed as the most efficient method due to its great ability to decrease the oil/water IFT from the high initial value (20−30 mN/m) to an ultralow value (10^{-3} mN/m) and to alter the wettability of the reservoir rock toward

the water-wet medium, which leads to a significant increase in oil recovery.[8] Moreover, the presence of surfactant can boost the formation of oil/water emulsion and improve interfacial rheological properties.[9] However, the fundamental issue during surfactant flooding is the loss of surfactant in the reservoir rock, which diminishes the surfactant flooding performance and its economic viability.[10] Reducing surfactant adsorption onto porous media is one of the most critical factors to evaluate the performance of surfactant flooding.[11]

To mitigate the surfactant adsorption density on reservoir rock, several studies have been conducted employing various additives such as alkalis and polymers in the last few years.[12,13] Generally, alkalis by creating negatively charged surfaces on the reservoir rock (mainly sandstone reservoirs), which causes a strong repulsive force between the anionic surfactant and porous media, and increasing the pH of the solution reduce surfactant adsorption.[14] However, the reaction of alkalis with reservoir rock

results in the formation of scaling ions, which leads to pore plugging and reduction in reservoir permeability. Furthermore, although polymers have been reported as sacrificial agents to decrease the surfactant adsorption,[12] high costs of polymers and their instability at high temperature and high salinity[15] impair their effectiveness in this matter. Therefore, the significant loss of surfactant in reservoir rock is still a principal problem that needs to be well addressed.

In recent years, nanoparticles (NPs) have been extensively employed in different EOR methods, such as CEOR, to investigate their potential for enhancing oil recovery.[16,17] In this regard, Rezaei et al.[18] observed that the IFT of SDS solution−oil was decreased by 77% (from 32.5 to 7.5 mN/m) in the presence of 0.05 wt % ZnO NPs. Similarly, Mohajeri et al. pointed out[19] that 100 ppm of ZrO$_2$ NPs can reduce a cationic surfactant−oil IFT from 18.4 to 5.4 mN/m. In another study, Wu et al.[20] demonstrated that the adsorption of an anionic surfactant on sand grains was greatly restrained by adding 5000 ppm SiO$_2$ NPs. In addition, Zargartalebi et al.[21] showed that hydrophobic silica NPs are more effective than hydrophilic silica NPs in reducing surfactant adsorption. Zhong et al.[22] investigated nonionic surfactant losses on Bakken and Berea rocks with and without SiO$_2$ NPs and observed a significant relationship between NP efficacy and the nature of adsorbents. Asl et al.[1] reported a 12.7% increase in oil recovery due to the synergistic effect between the amino acid surfactant and SiO$_2$ NPs. According to an investigation by Gbadamosi et al.,[23] aluminum oxide (Al$_2$O$_3$) polymeric nanofluid showed a better performance in viscosity increment, wettability alteration, and consequently oil recovery as compared to SiO$_2$ polymeric nanofluid. This is due to the stronger adsorption between Al^{3+} of Al$_2$O$_3$ and COO$^-$ of polymer. This is supported by Bashir Abdullahi et al.[24] who analyzed the potential of SiO$_2$, TiO$_2$, and Al$_2$O$_3$ NPs for EOR and obtained the highest oil displacement efficiency in the presence of Al$_2$O$_3$ NPs. To the best of our knowledge, no report has been found to date regarding the efficiency of surfactant−Al$_2$O$_3$ NPs as a chemical agent for CEOR.

This study therefore aims to experimentally investigate the capability of Al$_2$O$_3$ NPs to improve surfactant flooding performance for CEOR. For this purpose, Al$_2$O$_3$ NPs were dispersed within SDS surfactant at various concentrations of surfactant and NPs. The effect of Al$_2$O$_3$ NPs on surfactant properties including IFT and adsorption behavior onto sandstone rock was evaluated, and, in this regard, the main mechanisms of better efficiency were discussed. Furthermore, oil displacement experiments were conducted to determine the capability and suitability of SDS−Al$_2$O$_3$ nanofluid for CEOR.

2. EXPERIMENTAL SECTION

2.1. Materials. An anionic surfactant sodium dodecyl sulfate (SDS, purity 90%, mol wt 288.38 g/mol) bought from Merck was used in this work. Aluminum oxide nanoparticles (Al$_2$O$_3$, with 99% purity, size of 20−30 nm, specific surface area of 80 m^2/g) purchased from Skyspring Nanomaterials, Inc., Houston, TX., were used in this study to fabricate the nanofluid. Sodium chloride (NaCl) bought from Merk Group was used as an electrolyte to prepare synthetic reservoir brine. Crude oil from one of the Iranian oil fields was used as the hydrocarbon phase of the porous medium. The properties of the used oil in the experiment are shown in Table 1.

2.2. Preparation of Surfactant Solution. Initially, different amounts of SDS surfactant (e.g., 0.1, 0.15, 0.2, 0.25, 0.3, 0.4

Table 1. Properties of Used Crude Oil in the Experiment

viscosity (cp)	density (g/cc)	°API at 25 °C
26	0.885	29

wt %) were added to 100 mL of deionized water to obtain various concentrations of surfactant solution. Following this, a magnetic stirrer was applied to stir the solution, and stirring was stopped once a homogenous solution was obtained. The surfactant solutions were used in IFT, adsorption experiments, and the oil displacement efficiency test. The IFT method was employed to determine the CMC value of SDS. As presented in Figure 1, IFT is reduced by increasing the concentration of SDS until the inflection point of the curve; thus, 0.24 wt % SDS was determined as the CMC point, which is approximately consistent with the literature.[25] The interface of two fluids at the CMC point is fully covered by surfactant molecules, and there is no extra space for molecules; therefore, the addition of surfactant does not cause any remarkable reduction in IFT.

2.3. Preparation of the SDS−Al$_2$O$_3$ Nanofluid. To construct various nanofluids, Al$_2$O$_3$ NPs were first added to 100 mL of deionized water with different concentrations of NPs (e.g., 0.1, 0.2, 0.3, 0.4 wt %). The fluids were then shaken with an ultrasonic bath for 1 h to obtain a homogenized and stable suspension. Next, SDS powder at desired concentrations (e.g., 0.1, 0.15, 0.2, 0.24 wt %) were added to Al$_2$O$_3$ dispersion. The nanofluids were placed in a container and sealed to avoid contact with chemicals during the preparation process.

2.4. IFT Measurement. IFT between the oil phase and aqueous solutions was measured using an Easy Dyne Kruss Tensiometer K-20. All measurements were conducted at ambient temperature (25 °C) and atmospheric pressure employing the Du Nouy Ring method and the Harkins and Jordan ring correction method.

2.5. Static Adsorption Test. In this study, a batch technique was used to measure surfactant and surfactant nanofluid adsorption on sand grains.[21,26] To do so, first and foremost, the conductivity of the solutions was measured to assess appropriate calibration curves for each series of aqueous solutions.[27] Subsequently, surfactant and the surfactant nanofluid were placed in contact with the prepared sand particles with a ratio of 1:15 of sand grains and surfactant solution or nanofluid[22] (2 g of sand grains was mixed with 30 mL of fluid) in a horizontal vessel for 20 h (determined by an aging time optimization test) to ensure full interaction. Then, the surfactant solution and nanofluid are separated from sand samples by centrifugation at 2500 rpm for 15 min. After centrifugation, the concentration of fluids in the supernatant was measured employing appropriate calibration curves. The reason for using the conductivity method is that the concentration of solutions is independent of NP concentration.[21] By knowing the initial surfactant concentration and the equilibrium concentration, the adsorption density of surfactant can be calculated using eq 1[22]

$$q_e = \frac{V(C_i - C_{eq})}{m} \tag{1}$$

where q_e represents the adsorption density of surfactant (mg/g), V is the volume of the surfactant solution (L), C_i is the initial concentration of the solution (mg/L), C_{eq} is the surfactant concentration after being equilibrated with sand grains (mg/L), and m is the total mass of sandstone.

2.6. Oil Displacement Test. The influence of Al$_2$O$_3$ NPs on the efficiency of surfactant flooding for CEOR was studied by
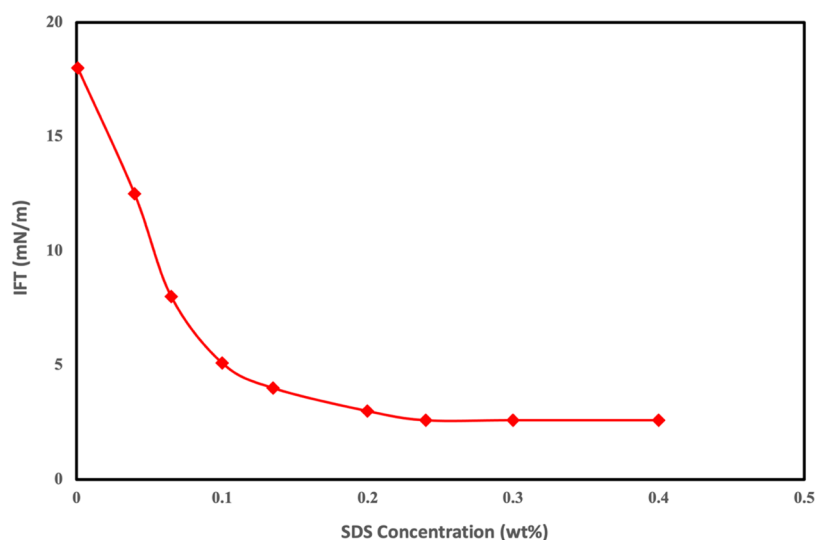
Figure 1. IFT value versus SDS concentration to determine the CMC point.

conducting sandpack flooding experiments. Sandpacks were loaded with sand grains in the range of 50−100 μm to represent a homogeneous porous medium. Table 2 shows the properties of

Table 2. Characteristics of Sandpacks

| | dimension (cm) | | | |
case	length	diameter	porosity (%)	permeability (mD)
water flooding	32.5	2.54	25.7	323
SDS solution flooding	31	2.51	24.2	301
SDS−Al$_2$O$_3$ nanofluid flooding	32	2.56	23.4	292

sandpacks for each flooding test. To prepare the sandpacks for the oil displacement test, sandpacks were vacuumed to remove

air trapped in porous media. Following this, sandpacks were saturated with prepared synthetic brine (3 wt %), and the crude oil was then injected to replace formation water until there was no water from the sandpack (S_{wc}). Formation water (3 wt % brine) was flooded at a constant flow rate of 0.25 cc/min until no more oil was observed in the effluent to evaluate the efficiency of water flooding. Moreover, SDS solution and SDS−Al$_2$O$_3$ nanofluid flooding were injected into sandpacks, which was continued until 2 pore volume (PV) was injected. All tests were conducted at ambient temperature (25 °C) and atmospheric pressure (Figure 2).

3. RESULTS AND DISCUSSION

3.1. Effect of Al$_2$O$_3$ NPs on IFT. As mentioned earlier, one of the CEOR functions is to decrease IFT between aqueous
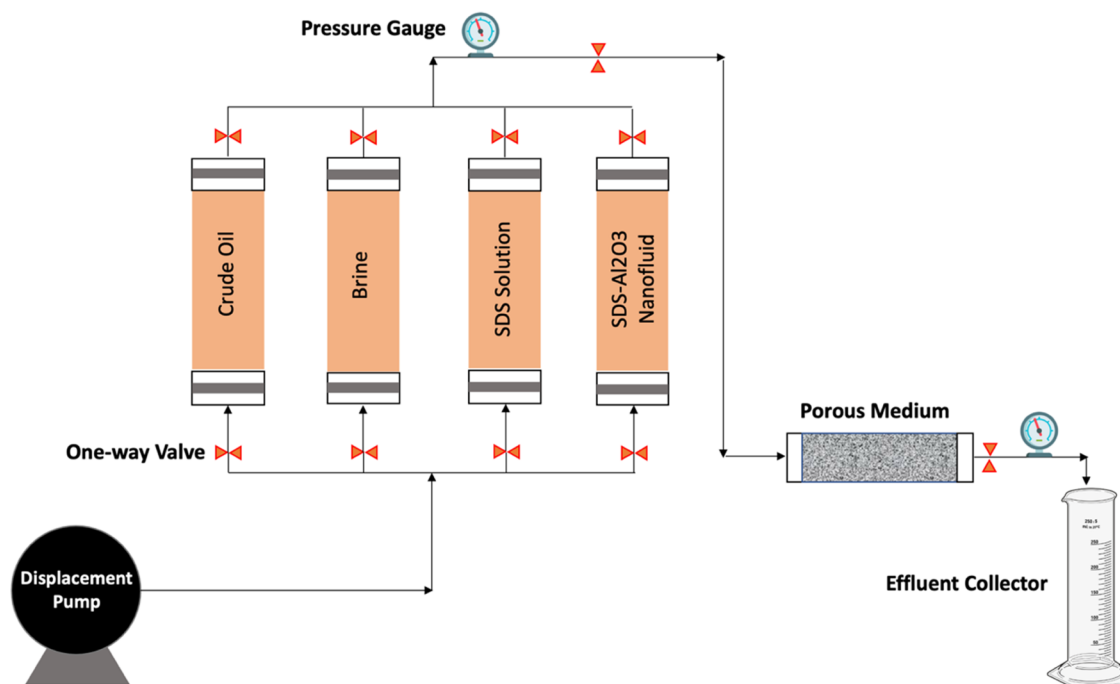

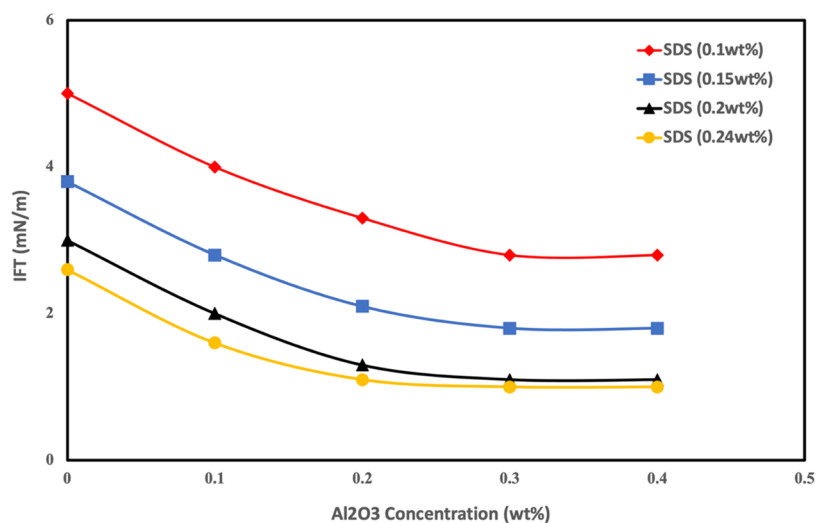**Figure 2.** Schematic of the apparatus for the oil displacement test.

Figure 3. IFT between the SDS solution and crude oil at different concentrations of Al_2O_3 NPs.
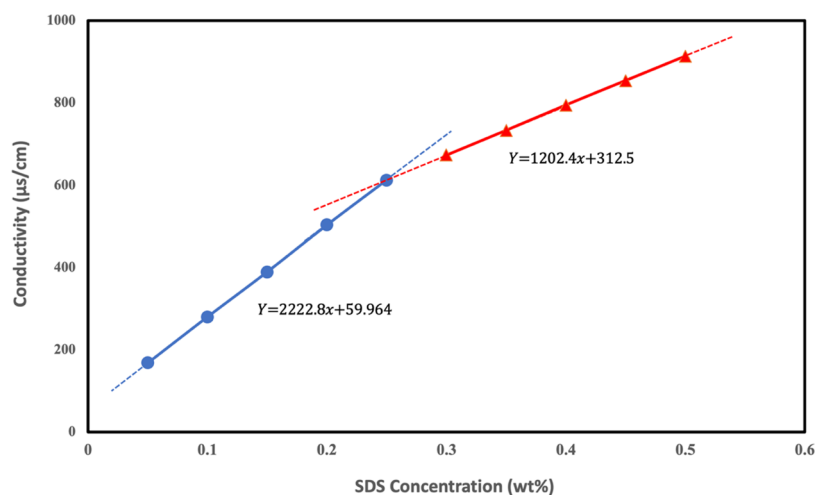

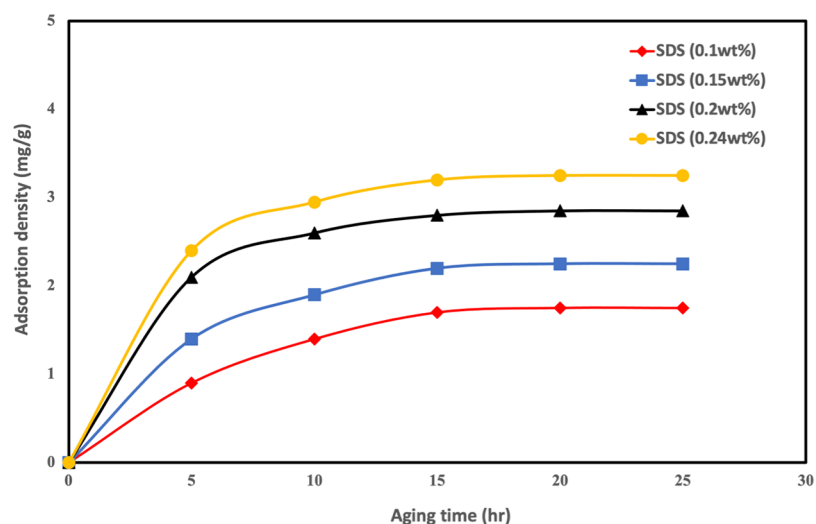**Figure 4.** Calibration curve for SDS concentration using the conductivity method.

Figure 5. Effect of aging time on surfactant adsorption.

phase and oil phase. The capillary number is a dimensionless quantity that represents the ratio between viscous forces and capillary forces (commonly around 10^{-7} for water flooding). A higher value of capillary number $(10^{-4}-10^{-2})$ means a lesser amount of residual oil saturation in the reservoir.[2] To achieve such a high number, the IFT needs to be decreased to an
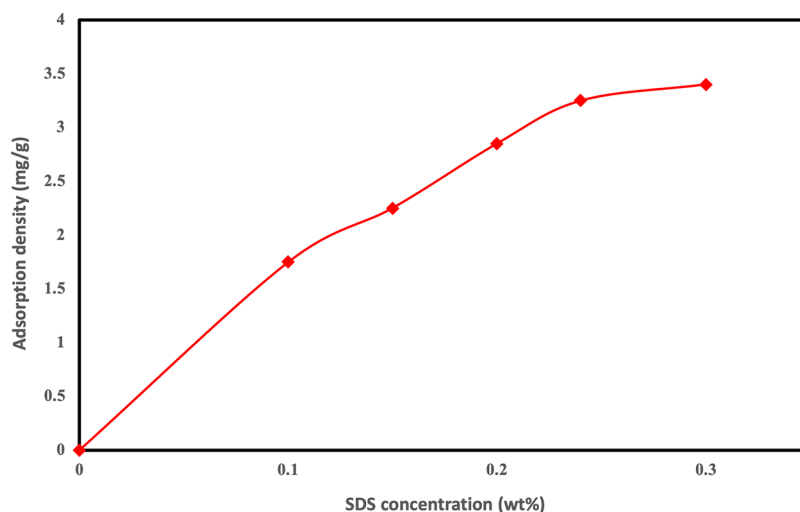
Figure 6. SDS adsorption on sandstone versus SDS concentration.

Table 3. Constants and Error Parameter of Two-Parameter and Three-Parameter Adsorption Isotherm Models[a]

| isotherm model	nonlinear form	linear form	parameters
Langmuir	$q_e = \dfrac{q_o K_L C_e}{1 + K_L C_e}$	$\dfrac{C_e}{q_e} = \dfrac{1}{K_L q_o} + \dfrac{C_e}{q_o}$	$q_o = 6.578$
			$K_L = 0.0004$
			$R^2 = 0.9576$
Freundlich	$q_e = b C_e^{1/n}$	$\ln q_e = \ln b + 1/n \ \ln C_e$	$n = 1.617$
			$b = 0.026$
			$R^2 = 0.9772$
Temkin	$q_e = \dfrac{RT}{b} \ln(K_m C_e)$	$q_e = \dfrac{RT}{b} \ln C_e + \dfrac{RT}{b} \ln K_m$	$b = 1603.16$
			$K_m = 0.0034$
			$R^2 = 0.9782$
Elovich	$\dfrac{q_e}{q_o} = K_e C_e e^{-(q_e/q_o)}$	$\ln \dfrac{q_e}{C_e} = \ln K_e q_o - \dfrac{q_e}{q_o}$	$q_o = 4.273$
			$K_e = 0.0006$
			$R^2 = 0.8659$
Redlich−Peterson	$q_e = \dfrac{K_r C_e}{1 + \alpha C_e^{\beta}}$	$\ln\left(\dfrac{C_e}{Q_e}\right) = \beta \ln C_e - \ln K_r$	$\beta = 0.3816$
			$K_r = 3.626$
			$\alpha = 137.48$
			$R^2 = 0.9423$

[a] q_e, q_o, and C_e are the equilibrium adsorption (mg/g), the maximum amount of surfactant adsorption (mg/g), and the adsorbate's equilibrium concentration (mg/L), respectively.

ultralow value $(10^{-3} \ \mathrm{mN/m})$.[28] Hence, IFT between SDS and crude oil in the absence and presence of Al_2O_3 NPs was measured to study the influence of NPs for further reduction in IFT. IFT reduction generally occurs when SDS and NPs are adsorbed at the fluid−fluid interface.[29]

Figure 3 illustrates IFT between the SDS solution and oil phase in the presence of Al_2O_3 NPs at different concentrations. As can be seen, the IFT decreased sharply on augmenting the concentration of NPs dispersed in SDS solutions, in which 0.3 wt % NPs reduced IFT by 76% when used with 0.1 wt % SDS. This significant reduction of IFT in the presence of NPs is mainly attributed to the ability of NPs to carry surfactant molecules by Brownian motion to the interfacial area.[30,31] As a matter of fact, the existence of extra surfactant molecules at the liquid−liquid interface diminishes the IFT value. According to the presented result in Figure 3, no IFT improvement was observed beyond 0.3 wt % Al_2O_3 NPs; a similar trend at all concentrations of SDS was observed. This can be explained by

the formation of NP aggregates at concentrations higher than 0.3 wt %, which disturbs the functioning of NPs as the carrier of surfactant molecules. Additionally, the negligible influence of NPs on IFT reduction was experienced at the CMC point. This is due to the saturation of the oil/water interface with surfactant molecules, and the existence of extra molecules is impossible at this point.[16] In general, the interaction of NPs and surfactants can lessen the IFT in the favor of CEOR.

3.2. Effect of Al_2O_3 NPs on Surfactant Adsorption. As mentioned earlier, the adsorption of surfactant onto reservoir rock is one of the most important parameters to evaluate the efficiency of surfactant flooding. Surfactant loss means a remarkable reduction of its concentration in chemical solutions and therefore an increase in oil/water IFT. As a result, the flooding process fails to meet its technical and economic objectives.[31,32] Generally, electrostatic interactions and van der Waals interactions between rock surfaces and surfactants are considered the main mechanisms of adsorption.[28,33]
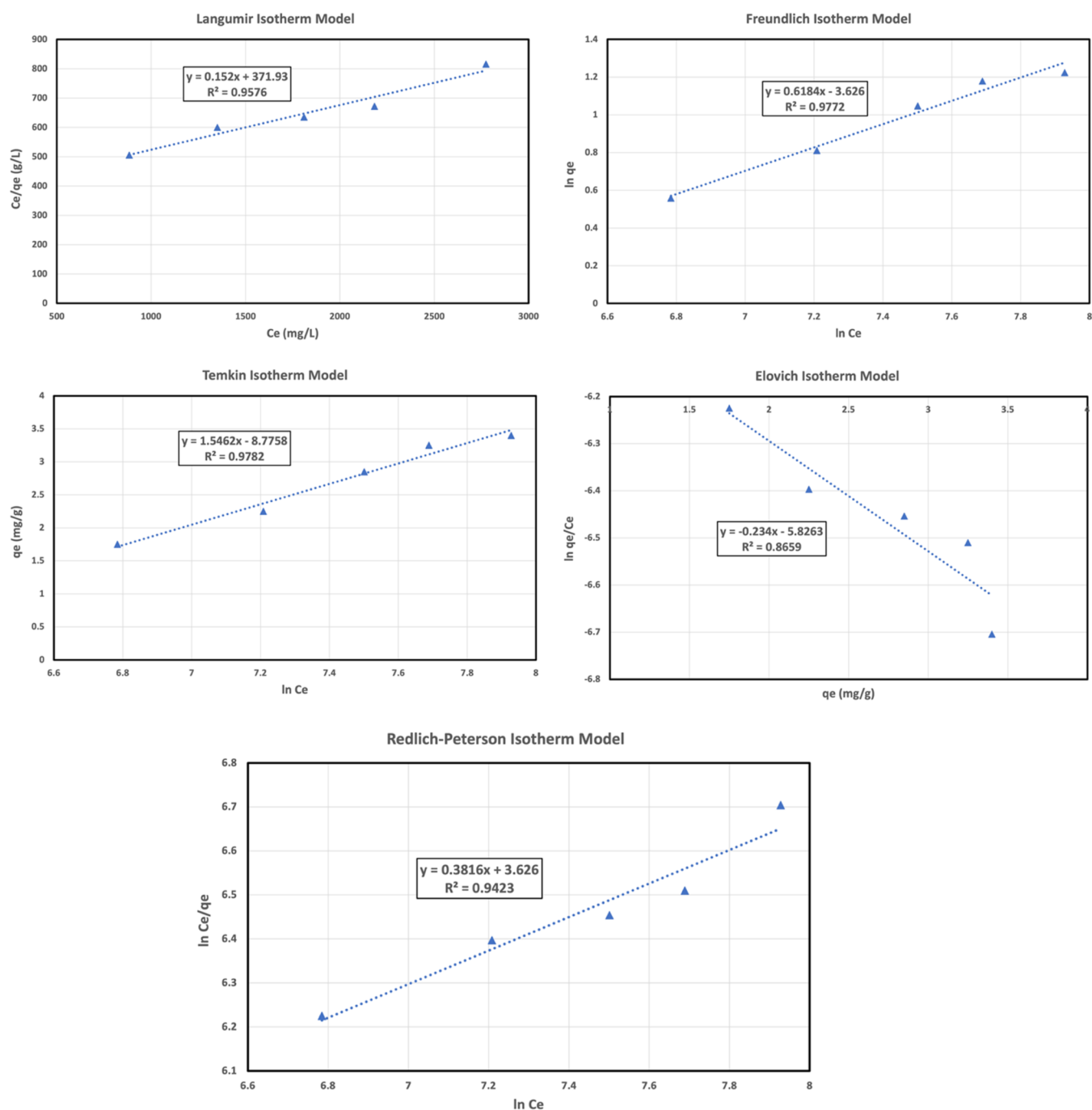
Figure 7. Langmuir, Freundlich, Temkin, Elovich (two-parameter), and Redlich−Peterson (three-parameter) adsorption isotherm fitting curves.

To measure the surfactant concentration after interaction with reservoir rock, the conductivity technique was used since solution conductivity is independent of NP concentration.[21] The conductance values were plotted against various surfactant solutions from 500 to 5000 ppm. Figure 4 shows the calibration curve of SDS concentration, which was used to analyze the adsorption behavior of surfactant onto sandstone rock in this study.

Prior to the static adsorption test, the effect of adsorption time on adsorption density was investigated to ensure that equilibrium was reached during experiments. The adsorption density of different concentrations of SDS was plotted against adsorption time in the range from 5 to 25 h. Figure 5 demonstrates the result of SDS adsorption against time. As can

be seen, in the early stage, SDS adsorption increased gradually until 15 h of aging time. Following this, there was a consistent change for 10 h, which means the adsorption of SDS onto sand grains reached equilibrium after 15 h. Accordingly, the experimented fluids (SDS solution and nanofluid) were placed in contact with sand grains for 20 h to ensure that equilibrium was reached during static adsorption tests. Figure 6 shows SDS adsorption density on sandstone against SDS concentration.

Table 3 and Figure 7 present the analysis of experimental adsorption data and calculated parameters and R^2 values for various two-parameter and three-parameter isotherm models, respectively. It is clear that the Temkin isotherm, which assumes a multilayer adsorption process, is a more appropriate model to fit experimental adsorption data compared to other models as
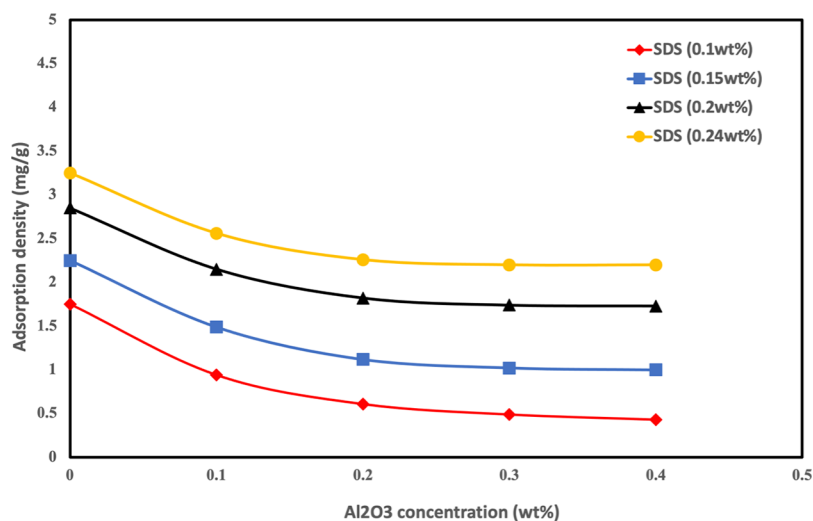
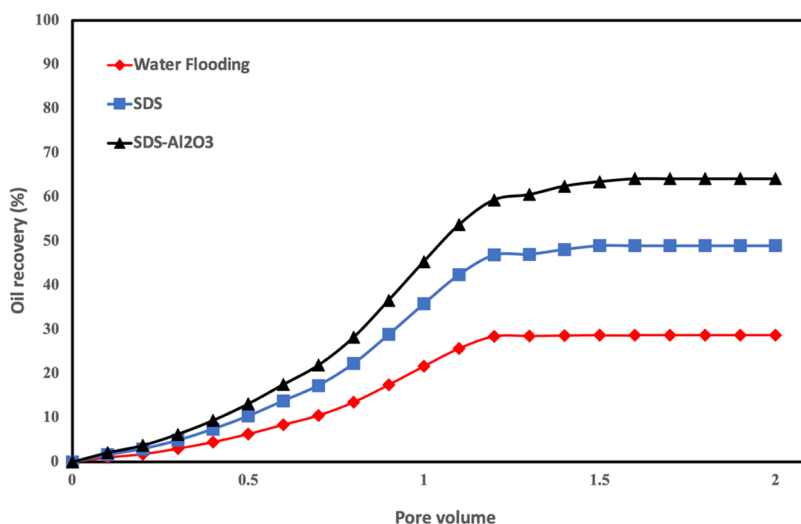**Figure 8.** Adsorption density of SDS versus Al_2O_3 concentration.

Figure 9. Oil recovery percentage versus pore volume injected for water flooding, surfactant flooding, and surfactant nanofluid flooding.

the R^2 value is higher than those of other isotherms. Although the Temkin isotherm is the best-fitted model, according to R^2 values, Langmuir and Freundlich models are desirable as well. A practical parameter associated with the Langmuir model is the separation factor (R_L), which determines the favorability of the adsorption process. R_L can be calculated using eq 2

$$R_L = \frac{1}{1 + K_L C_o} \tag{2}$$

where K_L is the Langmuir constant and C_o represents the highest initial concentration of surfactant.

Generally, $L_R < 1$ implies favorable, $L_R > 1$ shows unfavorable, and $L_R \sim 0$ and $L_R = 1$ indicate irreversible and linear adsorption processes, respectively. The values of R_L in this work are between 0.454 and 0.714, which shows that the adsorption of surfactant on sand grains is favorable. Additionally, a value of $0 < 1/n > 1$ in the Freundlich model shows the adsorption process is favorable. The value of $1/n$ in this study is 0.618 (consistent with findings of the R_L value in the Langmuir model).

The influence of NPs on the adsorption of varied SDS solutions on sandstone rock at varied concentrations was investigated. At the same concentration of the SDS solution, the adsorption density decreases considerably in the presence of NPs as presented in Figure 8. The adsorption of 0.1 wt % SDS was 1.76 mg/g in the absence of NPs, which decreased to 0.49 mg/g (72%) on adding 0.3 wt % Al_2O_3 NPs to the solution. Besides, the adsorption value was reduced from 3.25 to 2.2 mg/g (32.3%) in the presence of 0.3 wt % NPs at the CMC point. It was concluded that surfactant molecules prefer to be adsorbed on NPs instead of sand grains; in addition, the retained NPs may shield the sand wall, thus resulting in a significant reduction in surfactant adsorption.[28,34]

It is also important to mention that the effect of Al_2O_3 NPs in restraining the adsorption of SDS is negligible beyond 0.3 wt %, which can also be attributed to the formation of aggregates at concentrations higher than 0.3 wt %.[20] According to the IFT and adsorption test results, 0.3 wt % aluminum oxide NPs was selected as the optimum concentration of NPs in SDS solution. Overall, the adsorption of SDS on sandstone rock is largely preventable by employing NPs, which can increase the economic feasibility of surfactant flooding.

3.3. Flooding Test. Three flooding tests were conducted to evaluate the effectiveness of SDS–Al_2O_3 for CEOR and to compare it with SDS and water flooding. In this respect, the oil

recovery percentage versus the volume of injected fluid (water, SDS solution, SDS−Al$_2$O$_3$ nanofluid) is illustrated in Figure 9. As mentioned in the flooding test section, parameters such as the injected volume of fluid and the flow rate were the same during all flooding processes, and 2 PV of fluid was injected into the sandpacks. As presented in Figure 9, the highest oil recovery for brine (3 wt %) flooding was obtained at 1.5 PV, and no increase beyond 28.7% of OOIP was observed after this amount of fluid was injected into the sandpack. As the next step, 0.24 wt % SDS solution and SDS−Al$_2$O$_3$ nanofluids at an optimum concentration were flooded into sandpacks, and the results of these processes are shown in Figure 9. Greater oil recovery was observed for Al$_2$O$_3$ dispersion in SDS solution in comparison to the other flooding tests; the SDS solution in the presence of NPs caused 64.14% oil recovery, while this figure for SDS solution was 48.96%. The better performance of the SDS−Al$_2$O$_3$ nanofluid is due to the fact that NPs can greatly restrain the adsorption of surfactant onto reservoir rock. Besides, the friction caused by NPs on the adsorbent strips off the adsorbed molecules from the rock surface, hence reducing the surfactant adsorption.[20] Furthermore, NPs enhance IFT reduction, leading to a considerable improvement in the chemical flooding efficiency.

4. CONCLUSIONS

In this study, the capability of Al$_2$O$_3$ NPs to enhance surfactant efficiency was investigated. Prior to flooding experiments, the impact of nanoparticles on surfactant properties including IFT and adsorption behavior on sand grains was explored. The outcomes indicate a rapid decrease of IFT between aqueous phase and oil by adding Al$_2$O$_3$ NPs to the SDS solution. Moreover, surfactant adsorption onto sandstone rock was generally reduced in the presence of Al$_2$O$_3$ NPs, and this decrease was much more significant at higher concentrations of these NPs. Although the effectiveness of NPs gradually increases with an increase in their concentration, there is an optimal value of Al$_2$O$_3$ NP concentration. IFT and surfactant adsorption experiments revealed that beyond 0.3 wt % Al$_2$O$_3$ NPs, the effect of NPs is insignificant, and this concentration was selected for oil displacement experiments. Consequently, flooding tests showed that NPs can efficiently enhance the surfactant flooding performance and greater additional oil recovery can be obtained by adding NPs into the surfactant solution.

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