

## Study of adsorptive behavior of sulfonated polyacrylamide onto carbonate rock particles to enhance oil recovery

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**Abstract**—The adsorption of sulfonated polyacrylamide (SPAM) as an enhanced oil recovery polymer onto carbonate rock particles was studied using UV-visible spectroscopy. The effect of the experimental parameters of polymer concentration, salinity and temperature was investigated. The results showed that polymer adsorption increased as the SPAM concentration increased, but decreased as temperature and water salinity increased. Adsorption of the polymer in the presence of the anionic surfactant sodium lauryl ether sulfate (SLES) was evaluated in electrolyte media and in pure water. The adsorption equilibrium data for SPAM in electrolyte media was better fitted to the Langmuir isotherm than the Freundlich model. The thermodynamic parameters indicated that adsorption is a spontaneous and exothermic physisorption process.

Keywords: Adsorption, Carbonate Reservoir, Sulfonated Polyacrylamide (SPAM), Anionic Surfactant

### INTRODUCTION

Injection (flooding) of water into an oil well is a practical and effective way to maintain the reservoir pressure and sweep out oil in a more efficient manner. One problem with water flooding is that oil often has a higher viscosity than water, which may cause unstable displacement of oil. Increasing the viscosity of the injected water by adding a polymer will improve sweep during water flooding [1].

Polymer flooding is the most frequently used chemical enhanced oil recovery (EOR) method [2,3]. It makes the oil recovery process more efficient by decreasing water mobility. Water soluble polymers control mobility in reservoirs by increasing the viscosity of the injected water and, more importantly, by decreasing permeability through the adsorption and/or mechanical entrapment of the polymer molecules in the voids of porous media [4]. Adsorption of polymer onto reservoir rock decreases the polymer concentration in the aqueous phase, which decreases the viscosity and sweep efficiency of the injected polymer solution [4,5].

For optimal control of polymer viscosity at the desired reservoir locations, the adsorptive behavior of polymers on oil reservoir rock surfaces must be accurately assessed [6]. The adsorption of polymer onto a rock surface improves oil recovery by decreasing permeability in a highly permeable zone, particularly for heterogeneous reservoirs [7]. The adsorptive behavior of polymer on the reservoir rock must be known to determine its application in enhanced oil recovery. This can be done through a feasibility study of a polymer flooding project for optimization of oil recovery in

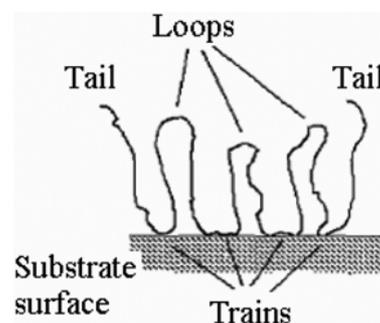


Fig. 1. Conformation of adsorbed polymer molecules at a solid/liquid interface [8].

consideration of the economic aspects of recovery.

The general conformation of adsorbed polymer molecules at the solid/liquid interface is shown in Fig. 1. The layers of polymers adsorbed onto the mineral surface consist of trains, loops and tails. Trains are segments of polymer molecule attached to the surface and are the bound section of the molecule acting as its anchor. The extended parts at both ends of the molecule are called tails. Between the trains are the loops which extend into the solution [8].

Partially hydrolyzed polyacrylamide (HPAM) is a copolymer of acrylic acid and acrylamide used extensively for EOR processes [9-11]. A number of studies [7,12-17] have examined the adsorption of polyacrylamide and its copolymers onto various carriers. Sabhapondit et al. [13] investigated the effect of pH and temperature on the adsorptive behavior of poly(N,N-dimethylacrylamide-co-Na 2-acrylamido-2-methylpropanesulfonate) copolymer onto the surface of sand. They found that adsorption is not as high as that of nonionic polymers or HPAM used in EOR. For this system, the adsorption is very low at neutral or basic pH and, at high

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temperatures, adsorption is low.

The effects of other variables on adsorption have also been studied. Hollander et al. [15] studied the adsorption of sulfonated polyacrylamides (SPAM) onto kaolinite at different pH values, ionic strengths and temperatures. They concluded that the adsorption of the polymer increased as the ionic strength increased but decreased as the pH decreased and that adsorption was independent of temperature. Rashidi et al. [16] examined the effect of the degree of sulfonation and the molecular weight on adsorption of SPAM polymers onto kaolinite and silica. They concluded that adsorption decreased as the degree of sulfonation increased while increasing the molecular weight led to higher adsorption.

Cheraghian et al. [17] studied the role of nanoparticles on the adsorption of water-soluble polymers onto solid surfaces of carbonate and sandstone. Shoaib et al. [18] investigated the effect of mineral type, salinity, background ions and temperature on adsorption of schizophyllan. They also studied the adsorptive behavior of the switchable surfactant (Ethomeen C12), which is a promising candidate for CO<sub>2</sub> foam to control mobility. Note that these studies differed in many respects from the present study, particularly for type of polymer, experimental conditions, type of surfactant, and type of reservoir used. The current study involves an Iranian oil reservoir.

Surfactant slugs are frequently used in EOR to mobilize residual oil by changing the wet ability of the rock or to decrease interfacial tension [19]. Surfactants are able to interact with polymer chains in solution and thus influence the corresponding rheological behavior. Many studies have examined the adsorption of polymers in the presence of surfactants [20-23].

Moudgil and Somasundaran [21] reported that the adsorption of an anionic polyacrylamide (APAM) was significantly affected by the charge characteristics of the surfactant. The presence of sodium dodecyl sulfonate (anionic surfactant) was found to decrease the adsorption of APAM onto hematite, but the presence of dodecyl amine hydrochloride (cationic surfactant) increased adsorption of the polymer. Siffert et al. [23] studied the adsorption of HPAM onto kaolinite in the presence of sodium dodecyl benzene sulfonate (NaDBS) and concluded that polymer adsorption was much

higher in a strong saline medium than in pure water. The addition of NaDBS decreased polymer adsorption in NaCl medium, but remained unaffected in CaCl<sub>2</sub> medium.

While the adsorptive behavior of different polymers onto various carriers has been studied, few reports have covered the adsorptive behavior of SPAM polymer onto carbonate rock, especially in electrolyte media at high temperature and in the presence of anionic surfactant. We prepared SPAM solutions in electrolyte media and examined the effect of polymer concentration, salinity and temperature on their adsorptive behavior. Because of the application of polymer-surfactant flooding in EOR, the effect of sodium lauryl ether sulfate (SLES), which shows high EOR efficiency, was studied as an anionic surfactant for the adsorptive behavior of SPAM polymer.

## EXPERIMENTAL

### 1. Materials

SPAM, having a 25% (mole) degree of sulfonation and an average molecular weight of  $8 \times 10^6$  Da, was obtained from SNF (France). SLES, having the chemical formula  $\text{CH}_3(\text{CH}_2)_{10}\text{CH}_2(\text{OCH}_2\text{CH}_2)_n\text{OSO}_3\text{Na}$  and 70% (wt) purity, was purchased from Kimiagaran Co. (Iran) and used to investigate the effect of surfactant on polymer absorption behavior. Carbonate particles with a mesh size of 16 produced from oil reservoir fields in southwestern Iran during oil production were used as the solid phase within adsorption tests.

### 2. Characterization of Reservoir Rock

X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopic (SEM) and x-ray fluorescence (XRF) were employed to characterize the samples. The FTIR spectra of reservoir rock particles are shown in Fig. 2. The rock has the characteristic peaks (876 and 712) of calcite rock, which confirms their nature [18]. The results of XRD analysis (Fig. 3) reveal that the reservoir rock is mostly calcite. SEM imaging (Fig. 4) shows the highly porous morphology of the reservoir rock with pores of different sizes and shapes and a particle size of less than 5  $\mu\text{m}$ . The chemical composition of the reservoir rock was obtained using XRF analysis and the results are presented in Table 1.

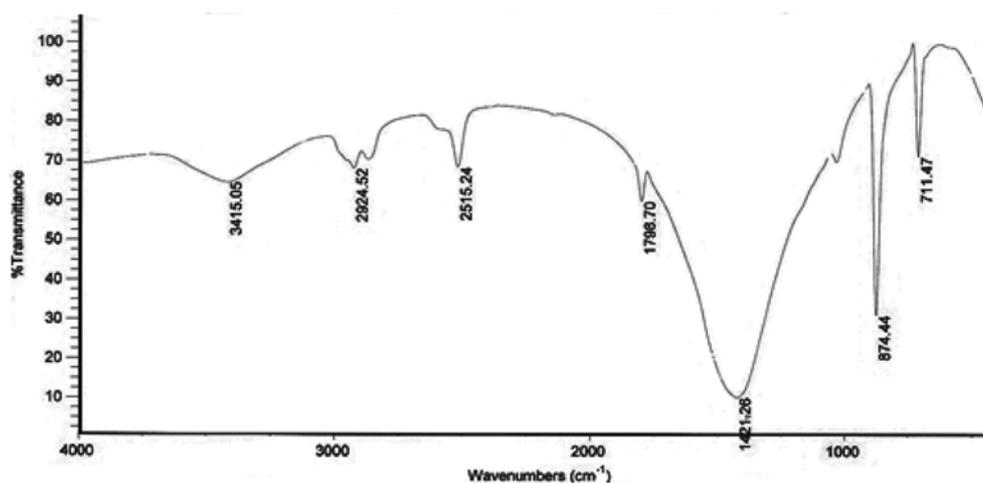


Fig. 2. FTIR spectra of reservoir rock.

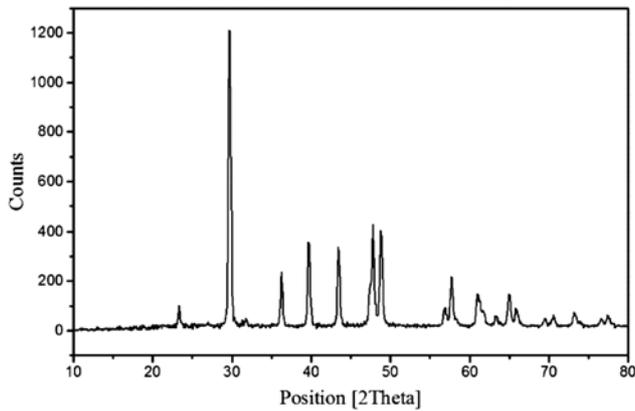


Fig. 3. XRD pattern of reservoir rock.

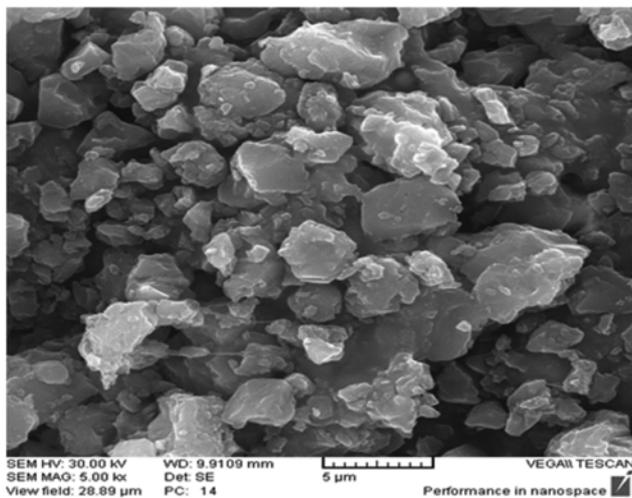


Fig. 4. SEM image of reservoir rock.

### 3. Adsorption Experiments

The SPAM solutions were prepared by the gradual addition of polymer powder to water having the desired salinity (Table 2) while being stirred to obtain a clear, homogeneous solution. The adsorbent (0.05 g carbonate particles) was poured into 10 mL polymer solution having different concentrations of SLES. The samples were immersed in a water bath at the desired temperature and were stirred slowly for different contact times. Their contents were then isolated by centrifugation (Model 3-30K; Sigma; Germany) for 30 min at 20,000 rpm.

Table 2. Composition of waters used

TDS (mg/L)	Ion concentrations (mg/L)										
	Ca <sup>2+</sup>	Mg <sup>2+</sup>	Na <sup>+</sup>	K <sup>+</sup>	HCO <sub>3</sub> <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	Cl <sup>-</sup>	NO <sub>3</sub> <sup>-</sup>	PO <sub>4</sub> <sup>3-</sup>	Li <sup>+</sup>	Al <sup>3+</sup>
1100	1470.2	-	-	-	-	-	-	-	-	-	-
5800	-	-	5844	-	-	-	-	-	-	-	-
6800	1470.2	-	5844	-	-	-	-	-	-	-	-
12900	790	213	3960	28	22	266	7300	49.8	11.1	1.1	<1
50800	2340	400	15150	240	312	290	30132	51	7.4	0.3	<1.1
232000	10000	2063	53000	1085	54	400	150662	534	-	2	<1

Table 1. Chemical composition of reservoir rock

Formula	wt%
L.O.I	43.6
Mgo	0.762
Al <sub>2</sub> O <sub>3</sub>	0.401
SiO <sub>2</sub>	0.567
P <sub>2</sub> O <sub>5</sub>	0.063
SO <sub>3</sub>	0.26
Cl	0.356
CaO	53.722
Fe <sub>2</sub> O <sub>3</sub>	0.227
Sr	0.041

The progress of adsorption was assessed by determining the residual concentration of polymer in the sample solution using a UV-visible spectrophotometer (Model: V-670; Japan) at  $\lambda_{max}=317$  nm. A series of standard solutions in which the polymer concentration was accurately known were prepared. The absorbance of each standard solution was measured and used to prepare a calibration curve showing how the experimental observable (absorbance) varied versus concentration. The Beer-Lambert law (Eq. (1)) states that the points on the calibration curve should yield a straight line:

$$A = \varepsilon L C_e \quad (1)$$

where A is absorbance,  $\varepsilon$  is the molar extinction coefficient ( $L \cdot \text{mole}^{-1} \cdot \text{cm}^{-1}$ ), L is the path length (10 mm), and  $C_e$  is concentration (mole/L) of the solution. The absorbance of each solution was measured after adsorption at the same wavelength, and the equilibrium concentration was calculated using the calibration curve. The polymer adsorption of adsorbent  $q_e$  (mg/g) was calculated using the following mass balance relation:

$$q_e = \frac{V(C_i - C_e)}{M} \quad (2)$$

where  $C_i$  (mg/L) and  $C_e$  (mg/L) are the initial and equilibrium concentrations of polymer, respectively, V (L) is the volume of the solution and M (g) is the mass of the adsorbent used.

## RESULTS AND DISCUSSION

The adsorption of polymer onto rock surfaces is strongly dependent on parameters such as temperature, pH, structure of the poly-

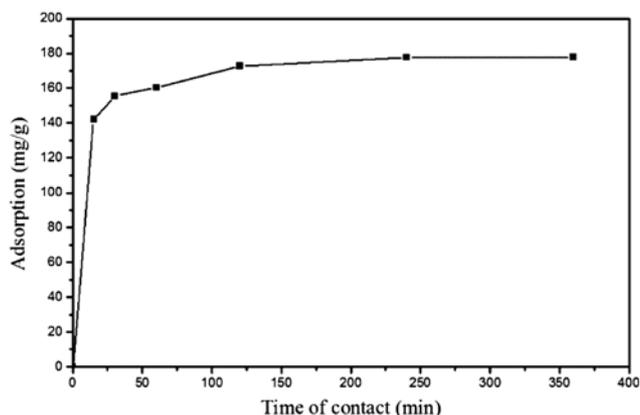


Fig. 5. Effect of contact time on the adsorptive behavior of SPAM polymer on carbonate particles at 90 °C, total dissolved solid (TDS)=6,800 mg/L and initial polymer concentration=2,000 mg/L.

mer and the surface, polymer concentration, contact time and salinity of the solvent. Some of these parameters are discussed below along with the experimental results.

### 1. Effect of Contact Time

The effect of time on the adsorptive capacity of SPAM is shown in Fig. 5. Approximately 90% of total adsorption occurred within the initial 15 min of contact time. It is clear that adsorption of the polymer was rapid, possibly because all sites on the surface were initially vacant and the concentration was high. As contact time increased, the number of adsorption sites decreased as a result of formation of an adsorbed polymer layer on the rock surface. When all sites were covered with polymer, further adsorption did not occur and time-independent adsorption occurred. The results indicate that as time progressed, polymer adsorption increased. After 4 h of contact time, the adsorption of SPAM remained constant over time; thus, a contact time of 4 h was chosen for the subsequent adsorption experiments.

### 2. Effect of Polymer Concentration

Zhang et al. [24] showed that the adsorption curve depends upon

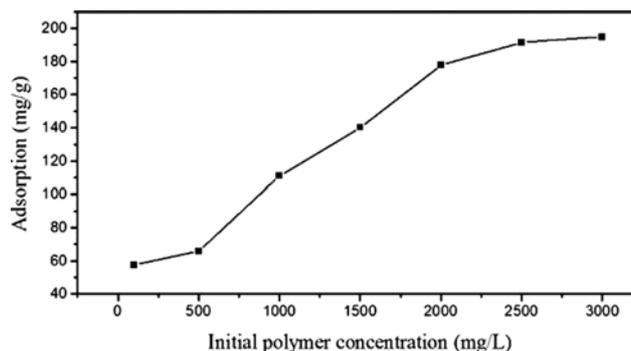


Fig. 7. Effect of polymer concentrations on the polymer adsorption at 90 °C and TDS=6,800 mg/L.

the concentrations in the dilute, semi-dilute and concentrated regimes (Fig. 6). In the dilute and concentrated regimes, polymer adsorption was basically concentration independent. In the semi-dilute regime, polymer adsorption was concentration dependent. The effect of SPAM polymer concentration on the adsorptive capacity of carbonate rock particles is shown in Fig. 7. This curve is similar to that observed by Zhang et al. In the dilute regime (SPAM < 500 mg/L), the polymer molecules appear as a free coil in solution and take a flat orientation on the rock surface during adsorption. In the semi-dilute regime (500 mg/L < SPAM concentration < 2,000 mg/L), some polymer molecules were adsorbed with all their segments in contact with the surface and some were partially in contact. The increase in concentration increased total adsorption.

In the third regime (SPAM concentration > 2,000 mg/L), one end of the polymer attached to the rock surface while the majority of molecules were free in solution and all adsorption sites were filled, making the adsorption concentration independent. At higher concentrations of polymer solution, the number of polymer molecules increased, which increased the probability of interaction between the rock surface and the polymer molecules. As the ionic copolymers of acrylamide in electrolyte media were adsorbed onto the reservoir rock particles, electrostatic forces contributed to the interaction between the polymers and the surface. The adsorption of an

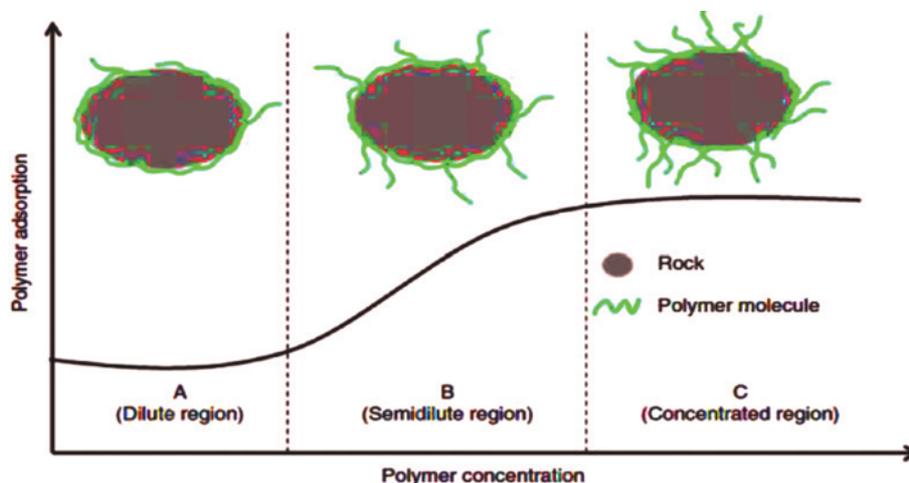


Fig. 6. Proposed polymer adsorption mechanism by Zhang et al. [24].

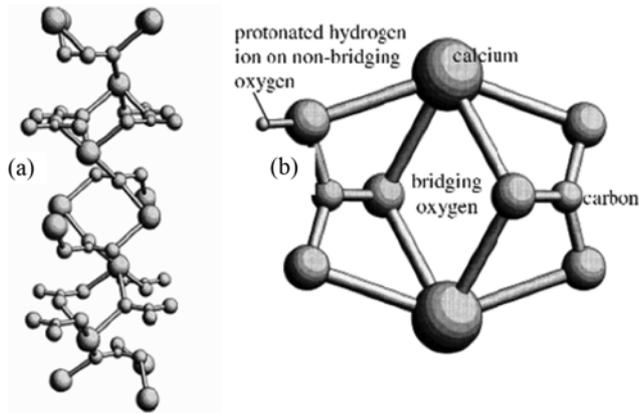


Fig. 8. (a) Lattice molecular of calcium carbonate and (b) surface structure of calcium carbon [25].

anionic polymer at the solid-liquid interface is strongly influenced by the charged groups on the surface.

Carbonate rock is a potential reservoir rock composed primarily of calcium carbonate. Fig. 8(a) shows the 3D structure of calcium carbonate, which explains only the bulk structure. The surface structure of carbonate rock differs from its bulk structure. Oxygen atoms determine the surface charge of carbonate rock. Mao and Siders [25] used the Hartree-Fock model to describe the surface structure and charge of calcium carbonate dimers.

In the Hartree-Fock model, oxygen atoms can be categorized as non-bridging (bonding with one calcium atom) or bridging (bonding with two calcium atoms). On the stabilized structure, the non-bridging oxygen atoms represent the edge of the surface (or surface structure). The protonation of hydrogen ion occurs at the non-bridging oxygen atoms, resulting in a positive charge at the surface. The stabilized rotation of calcium carbonate dimers is shown in Fig. 8(b). Adsorption of SPAM onto the surface of the carbonate rock was significantly high at this pH. This can be attributed directly to the attractive electrostatic interaction between the negatively charged sulfonated groups of polymer and the positive surface of the carbonate rock, which increased the adsorption of SPAM onto the reservoir rock particles.

After a certain concentration, no adsorption took place because of the saturation of the adsorptive capacity of the active adsorption sites. In this study, adsorption saturation was achieved at the initial polymer concentration of 2,000 mg/L.

### 3. Effect of Salinity

The addition of salts to polymer solution affects conformation of the polymer chains from changes at the electrical double layer around the polyions. As the salt content increases, the double electrical layer on the polymer molecular chains are compressed and the electrostatic repulsion among the anions is shielded, decreasing the hydrodynamic radius of the polymer molecules [26-28]. The adsorption of the polymer should be influenced by the salinity of the water.

Fig. 9 shows the effect of total dissolved salt (TDS) in the water on the adsorption of SPAM solution onto the carbonate rock particles. The adsorptive capacity of the polymer decreased as the TDS increased. This appears to be associated with changes in the param-

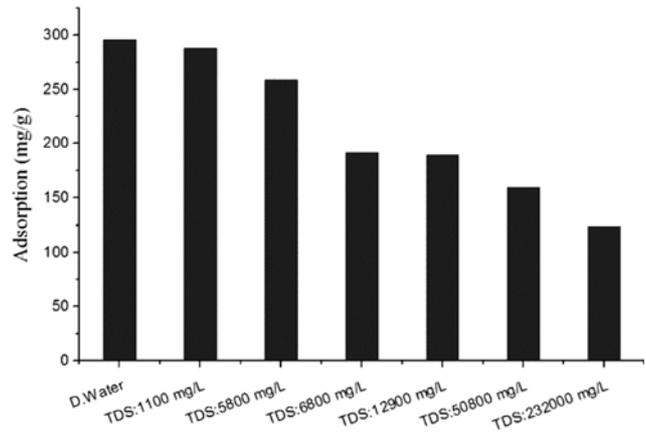


Fig. 9. Effect of total dissolved salt (TDS) in the water on the adsorption of SPAM solution at 90 °C and initial polymer concentration=2,500 mg/L.

eters of the electrical double layer on the polymer chain. When the SPAM solution adsorbed onto the carbonate rock particles, the SPAM molecules constituted the larger number of contacts with the surface because of electrostatic interaction. An increase in salinity of the polymer solution screened the anionic sulfonated groups of the SPAM chain with the cations of the salts and decreased the electrostatic interaction between the polyelectrolyte and the surface. This resulted in a decrease in SPAM adsorption onto the carbonate rock particles with the increase in TDS.

Theoretically, when the electrostatic forces between the adsorption surface and adsorbate ions are attractive, as in this system, an increase in salt concentration will decrease the adsorptive capacity. Conversely, when the electrostatic attraction is repulsive, an increase in salt concentration will increase adsorption [12,13,29,30].

### 4. Effect of Temperature

The nature of adsorption of SPAM onto carbonate rock particles was studied at 303 K, 323 K, 343 K and 363 K (Figs. 10 and 11). Fig. 10 shows that adsorptive capacity decreased as the solution temperature increased from 303 K to 363 K, indicating that adsorption of SPAM onto carbonate rock particles was exother-

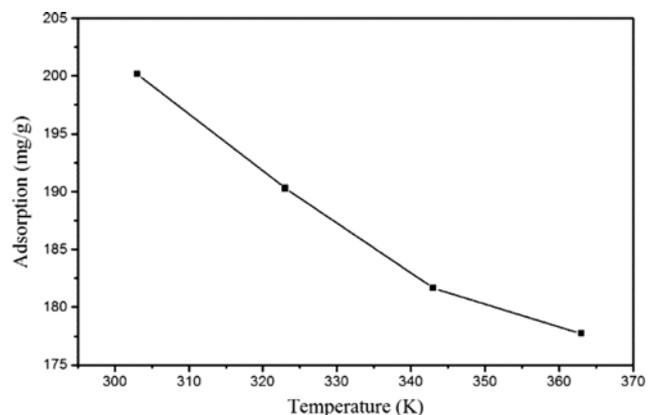


Fig. 10. Effect of temperature on the adsorption of SPAM solution at 90 °C, TDS=6,800 mg/L and initial polymer concentration=2,000 mg/L.

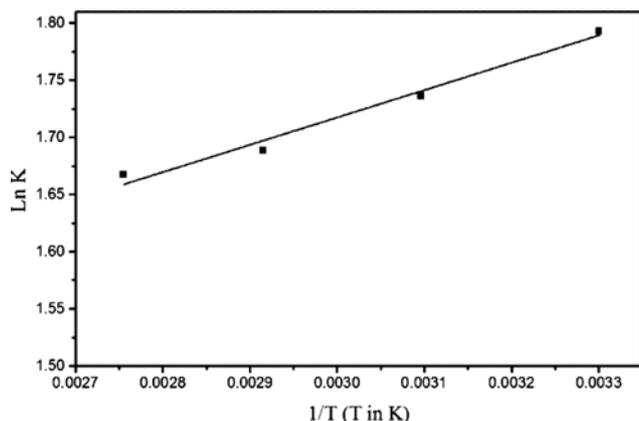


Fig. 11. Plot of Ln K versus 1/T (T in Kelvin).

mic and the amount of adsorbed polymeric chains decreased as the temperature increased [13]. This could be attributed to the weakening of attractive forces between the adsorbate and the adsorbent.

To learn more about the adsorptive behavior of SPAM polymer onto carbonate rock particles, the thermodynamic parameters of enthalpy ( $\Delta H$ ), entropy ( $\Delta S$ ) and Gibbs free energy ( $\Delta G$ ) were evaluated using the following equations:

$$K_C = \frac{C_{Ae}}{C_e} \quad (3)$$

$$C_{Ae} = C_t - C_e \quad (4)$$

$$\Delta G = -RT \ln K_C \quad (5)$$

where  $K_C$  is the equilibrium constant and  $C_{Ae}$  (mg/L) and  $C_e$  (mg/L) are the equilibrium concentrations for the solute on the sorbent and in solution, respectively. Parameters  $\Delta H$  and  $\Delta S$  are determined using the van't Hoff equation as a function of temperature as:

$$\ln K_C = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (6)$$

Parameters  $\Delta H$  and  $\Delta S$  were obtained from the slope and intercept, respectively, of the plot in Fig. 11. The change in  $\Delta G$  for the adsorption of SPAM was  $-4.516$ ,  $-4.662$ ,  $-4.815$  and  $-5.032$  kJ/mole at 303 K, 323 K, 343 K and 363 K, respectively. The negative values for  $\Delta G$  resulted from the spontaneous nature of adsorption, which is thermodynamically favorable at the temperatures under investigation (303 K to 363 K). The value of  $\Delta H$  was  $-1.952$  kJ/mole, which indicates that adsorption here is exothermic.

Physical adsorption and chemisorption can be classified, to a certain extent, by the magnitude of the change in enthalpy. Physisorption is associated with enthalpy of adsorption values greater than  $-25$  kJ/mole, whereas values smaller than  $-40$  kJ/mole are typical of chemical adsorption [13]. Based on this, the adsorption of SPAM onto carbonate rock particles is assumed to be a physical adsorption process.

The value of  $\Delta S$  was  $0.0084$  kJ·mole $^{-1}$ ·K $^{-1}$ . A positive value for  $\Delta S$  signifies that randomness in the solid-liquid interface increased throughout adsorption.

### 5. Effect of Presence of Anionic Surfactant

It is known that surfactant-polymer (SP) systems are used in EOR

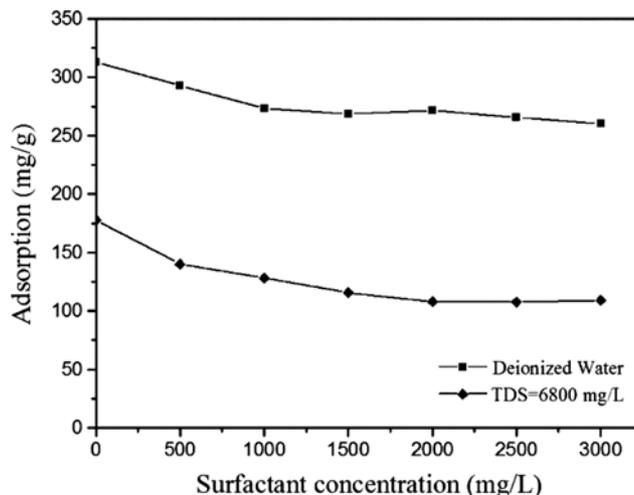


Fig. 12. Effect of SLES on the adsorption of SPAM solution in electrolyte with TDS=6,800 mg/L and deionized water at 90 °C, initial polymer concentration=2,000 mg/L.

because of their high EOR efficiency [31]. The addition of surfactants to polymer solution lowers the interfacial tension between the injected polymer solution and the reservoir oil [4]; however, the presence of a surfactant can affect the adsorption of the polymer onto the surface of the reservoir rock. To investigate the effect of a surfactant on the adsorption of SPAM polymer onto the surface of reservoir rock, SLES was selected because it is commonly used as an anionic surfactant in EOR applications.

Fig. 12 shows the adsorptive capacity of SPAM solution onto the carbonate rock particles as a function of SLES concentration in deionized water media and electrolytes (TDS=6,800 mg/L). These results indicate that adsorption of SPAM in deionized water and electrolyte media decreased as the surfactant concentration increased. The interaction between the polymer and the surfactant can influence adsorption of the polymer. Polymer and surfactant are either positive or negative; hence, binding of SLES to SPAM is prevented by the strong electrostatic repulsion between the anionic groups of the polymer and the polar heads of the surfactant molecules. Changes in the conformation of the polymer chains in the presence of anionic surfactant caused by screening of the electrostatic repulsion between groups of polymer charged by the counter-ions of the surfactant species produced significant changes in polymer adsorption [15, 32]. Both SPAM and SLES molecules can adsorb onto the surface of carbonate rock particles. Competition for the positive sites on the carbonate rock between similarly charged SPAM and SLES may be responsible for decreased adsorption of SPAM in the presence of SLES [33]. On the other hand, the surface area available for adsorption by the polymer decreased in the presence of surfactant molecules on the solid surface. Both of these factors appear to contribute to the decrease in adsorption of SPAM onto the rock reservoir in the presence of SLES.

### 6. Adsorption Isotherms

Adsorption isotherms are usually used to describe a sorption system. Adsorption data is generally interpreted using the Langmuir and the Freundlich isotherm models. The Langmuir model assumes a structurally homogeneous adsorbent where all adsorp-

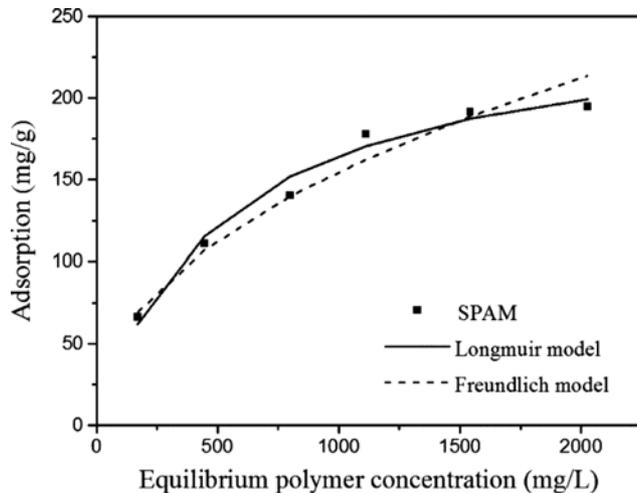


Fig. 13. Adsorption isotherms of SPAM on carbonate rock particles at 90 °C, TDS=6,800 mg/L and initial polymer concentration=2,000 mg/L.

tion sites are identical and energetically equivalent. The Freundlich model describes a heterogeneous system characterized by a heterogeneity factor of  $1/n$ . This model describes reversible adsorption and is not restricted to the formation of the monolayer. The Langmuir and the Freundlich models are shown in Eqs. (7) and (8), respectively [34]:

$$q_e = \frac{Q_m K_L C_e}{1 + K_L C_e} \quad (7)$$

$$q_e = K_f C_e^{1/n} \quad (8)$$

where  $q_e$  (mg/L) is the amount adsorbed at equilibrium,  $C_e$  (mg/L) is the equilibrium concentration of polymer in solution;  $Q_m$  (mg/g),  $K_L$  (L/mg),  $K_f$  ( $\text{mg}^{1-n} \cdot \text{L}^n/\text{g}$ ) and  $n$  are isotherm constants, for maximum adsorptive capacity, energy of adsorption, adsorptive capacity and adsorption intensity, respectively.

The results for fitting of the Langmuir and Freundlich isotherms are plotted in Fig. 13. The isotherm constants for both models were determined by linear regression of the experimental data. Table 3 lists the correlation coefficients ( $R^2$ ). The results indicate that the adsorption equilibrium data for SPAM solution at 90 °C and in electrolyte media (TDS=6,800 mg/L) is best fitted to the Langmuir isotherm model ( $R^2=0.99$ ) and signifies monolayer coverage of the polymer onto the surface of the carbonate rock particles [35].

## CONCLUSION

The present study investigated the adsorptive behavior of SPAM solution onto carbonate rock particles at high temperature. It was

Table 3. Values of the Langmuir and Freundlich constants for the adsorption of SPAM solution at 90 °C, TDS=6,800 mg/L

Langmuir			Freundlich		
$Q_m$ (mg/g)	$K_L$ (L/mg)	$R^2$	$n$	$K_f$ ( $\text{mg}^{1-n} \cdot \text{L}^n/\text{g}$ )	$R^2$
250	0.002	0.990	2.192	6.622	0.976

found that the adsorption of SPAM solution was affected by polymer concentration, temperature and salinity. Polymer adsorption increased as the SPAM content increased and decreased as the temperature and water salinity decreased. The experimental results also showed that the addition of SLES as an ionic surfactant decreased polymer adsorption in electrolyte media and in pure water at high temperature by decreasing the surface area available for adsorption of the polymer. The Langmuir model was found to be a more suitable fit for the experimental data than the Freundlich model. Thermodynamic study demonstrated that the SPAM adsorption is a spontaneous and exothermic process.

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