

Adsorption Behavior of Poly(*N,N*-dimethylacrylamide-*co*-Na 2-acrylamido-2-methylpropanesulfonate) on Sand Surface

Anupom Sabhapondit,¹ Arun Borthakur,¹ Inamul Haque²

¹Regional Research Laboratory, Jorhat 6, Assam, India

²Dibrugarh University, Dibrugarh 4, Assam, India

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ABSTRACT: The adsorption behavior of poly(*N,N*-dimethylacrylamide-*co*-Na 2-acrylamido-2-methylpropanesulfonate), an enhanced oil recovery polymer, was studied. Adsorption isotherms show that adsorption on bentonite is very high followed by that on limestone that, in turn, is much higher than that on sand surface following the order: bentonite \gg limestone $>$ sand. On the addition of NaCl, adsorption on sand surface decreased to a minimum value and then increased. Adsorption increased with decreasing pH and also decreasing the content of Na 2-acrylamido-2-

methylpropanesulfonate in the copolymer. The amount of the copolymer adsorbed on the sand surface is comparable to that of partially hydrolyzed polyacrylamide but much less than that of poly(acrylamide-*co*-vinylpyrrolidone). The type of adsorption was found to be physical, which is supported by the enthalpy of adsorption as well as by IR spectra. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 91: 2482–2490, 2004

Key words: polyelectrolytes; adsorption; turbidimetry; enhanced oil recovery; physisorption

INTRODUCTION

Copolymers of *N,N*-dimethylacrylamide with sulfonated comonomers, especially the Na salt of 2-acrylamido-2-methylpropane sulfonic acid, is one of the promising agents for the polymer flooding technique of oil recovery.^{1–4} The main advantages of this polymer over others are as follows: (i) it is thermally stable at least at 120°C for a long period of time; and (ii) it is brine compatible; that is, it does not precipitate in the presence of bivalent ions even at 120°C.³ A few other thermally stable copolymers have been reported in the literature that are also brine compatible.^{5–7} The most widely used synthetic enhanced oil recovery (EOR) polymer, partially hydrolyzed polyacrylamide (PHPA) suffers excessive hydrolysis at high temperature and consequently precipitates in the presence of bivalent cations present in the reservoirs.^{5–7} However, with increasing demand of petroleum in today's world, recovery of residual oils from the deeper and consequently hotter reservoirs becomes essential. Therefore, polymers suitable for such reservoirs become increasingly more important for the oil industry.

Water-soluble polymers improve oil recovery by increasing the sweep efficiency of the aqueous phase. This can be achieved by decreasing the mobility of the aqueous phase or by increasing the viscosity of the

aqueous phase.^{8,9} Adsorption of polymer on reservoir rock causes concentration depletion of polymer in the aqueous phase and hence reduction of viscosity. Excessive adsorption of a polymer may make the use of the polymer uneconomic for EOR application.^{8,10} Therefore it is important to study the adsorption behavior of a polymer before it is finally used in the field. In the present work, the adsorption behavior of poly(*N,N*-dimethylacrylamide-*co*-Na 2-acrylamido-2-methylpropanesulfonate) (NNDAM–NaAMPS) was studied under different conditions of salinity, temperature, pH, and polymer concentrations. The study was performed mostly on sand surfaces, although adsorption on bentonite and limestone surfaces was also determined for comparison. Adsorption of PHPA, which is anionic, and poly(acrylamid-*co*-*N*-vinylpyrrolidone) (AM–NVP), which is a nonionic polymer, were also measured for a comparative study.

EXPERIMENTAL

Synthesis and characterization of polymers

Monomers used for preparing the copolymers were *N,N*-dimethylacrylamide (NNDAM) of Aldrich Chemicals (Milwaukee, WI); acrylic acid (AA) of G. S. Chemicals; 2-acrylamido-2-methylpropanesulfonic acid (AMPS), acrylamide (AM), and *N*-vinylpyrrolidone (NVP) of Merck-Schuchardt (Darmstadt, Germany). NNDAM was exposed over calcium hydride for 24 h to remove stabilizer and then distilled under vacuum. AMPS was purified by recrystallization from ethanol.

Correspondence to: A. Borthakur (arunabth@rediffmail.com).

TABLE I
Characteristics of the Copolymers

Sample	Copolymer type (A-B)	F_b^a		Intrinsic viscosity (dL/g) at 25°C in 0.1M NaCl	MW ($\times 10^{-6}$)	
		F_{bp}^b	F_{bs}^c		M_n	M_w
1	NNDAM–NaAMPS	49	—	5.78	—	—
2	NNDAM–NaAMPS	41	—	5.31	—	—
3	NNDAM–NaAMPS	37	42	5.40	0.33	1.49
4	NNDAM–NaAMPS	30	—	5.52	—	—
5	NNDAM–NaAMPS	16	—	5.12	—	—
6	AM–NaAA	30	—	14.40	—	—
7	AM–NVP	—	34	5.34	—	—

^a Wt % of B in copolymer.

^b Determined potentiometrically.

^c Determined from ¹H-NMR.

AM was recrystallized from methanol. AA and NVP were used as received. Ammonium persulfate and sodium metabisulfite were obtained from CDH (New Delhi, India) and purified by recrystallization from ethanol. Azobisisobutyronitrile (AIBN) was obtained from Otto Kemi and used after recrystallization from methanol.

Synthesis of acrylamide copolymers is a well-established phenomenon.¹¹ NNDAM–NaAMPS and AM–NaAA were prepared by aqueous solution polymerization technique using persulfate/metabisulfite redox pair as initiator. The monomers were dissolved in degassed, distilled water to give a 12 wt % solution in a three-neck round-bottom flask. Nitrogen gas was bubbled through the reaction mixture for 1 h at 25°C with slow stirring. Then 0.15% (by wt of monomer) ammonium persulfate and 0.2% (by wt of monomer) sodium metabisulfite in solution were added dropwise to the reaction mixture. The reaction flask was kept at a thermostatic bath at 25°C for 6 h providing constant stirring to the solution. With the formation of the polymer, the reaction mixture became highly viscous. A small portion of the reaction mixture was precipitated from acetone, dried in a vacuum oven at 45°C for 15 h, and kept for potentiometric titration. The other part was diluted to 3 times its volume and neutralized with 2% NaOH solution. Then it was precipitated from acetone and dried in a vacuum oven at 45°C for 15 h.

AM–NVP was prepared by aqueous solution polymerization using AIBN as the initiator. AM and NVP were dissolved in degassed, distilled water to give a 25 wt % solution. Nitrogen was bubbled through the solution for 1 h. Then the reaction mixture was warmed to 60°C and 0.075% (by wt of monomer) AIBN was added to it and mixed thoroughly by gentle stirring. The temperature was maintained at 60°C by a constant temperature bath. Polymer formed within 0.5 h. The reaction mixture was diluted to 3 times its original volume with distilled water and a slight amount of hydroquinone was added for terminating

the polymerization. The polymer was precipitated from acetone and dried in a vacuum oven at 45°C for 15 h.

All the copolymers were characterized for intrinsic viscosity and compositions.^{12–15} The presence of functional groups was confirmed from the IR and ¹H-NMR spectra. Compositions of all the ionic copolymer samples were determined potentiometrically. ¹H-NMR spectra were also used to determine the composition of some samples. The molecular weight and molecular weight distribution of sample 3, which was used as the main sample for investigation, were determined. Characteristics of the copolymers used are presented in Table I. A more detailed characterization of the polymers is available elsewhere.³

Adsorbents used

In all cases, the sand used was washed with hot, concentrated HCl and then washed repeatedly with distilled water to remove the traces of HCl. The sand was then dried in an oven at 120°C for 1 week. Finally, it was sieved with meshes of proper sizes. Physical characteristics of the sand used are presented in Table II. The limestone used was crushed well and sieved with BS-72 mesh. Bentonite supplied by Loba Chemicals was used as received.

Other chemicals used

Reagent-grade acetone was obtained from S.D. Fine Chemicals (Mumbai, India). Sodium citrate was ob-

TABLE II
Physical Characteristics of the Sand Used

Physical property	Numerical value
Diameter at 10% (μm)	128.09
Diameter at 50% (μm)	196.45
Diameter at 90% (μm)	405.42
Mean diameter (μm)	226.14
Density (g/cm^3)	2.6177

tained from IDP Ltd. and NaCl was from Merck. Hyamine-1622 was obtained from BDH (Poole, UK). All these chemicals were used without further purification.

Instrumentation

Kinematic viscosities were measured by an Ubbelohde viscometer (Cannon-Ubbelohde, State College, PA) fitted with a Schott Gerate AVS 400 automatic instrument and a thermostatic bath. IR spectra were taken in KBr pellets in a Perkin-Elmer FTIR system 2000 (Perkin Elmer Cetus Instruments, Norwalk, CT). $^1\text{H-NMR}$ spectra of 10 wt % polymer solution were taken in a Bruker 300 MHz instrument (Bruker Instruments, Billerica, MA) using TMS as the reference and D_2O as the solvent. Polymer molecular weight was determined by a Waters GPC (Waters Chromatography Division/Millipore, Milford, MA) at 25°C equipped with an RI detector and using Shodex OH Pak columns KB 802.5, 803, 804, and 806. Polyacrylamide was used as a calibrant and 0.2M NaNO_3 was the eluent. Absorbance was determined by a Chemito UV-visible spectrophotometer using a 10-mm quartz cell. Potentiometric titration was carried out in a Systronics mV and pH meter equipped with a pair of glass and calomel electrodes. Size and distribution of sand particles were determined by a Cilas particle-size analyzer. Density of sand was determined by a liquid displacement method using a specific gravity bottle. An electronic Mettler balance (Mettler, Greifensee, Switzerland) sensitive up to $1/10,000$ th of a gram was used for all weighing purposes. A Julabo F32 bath was used to maintain constant temperatures during reactions.

Preparation of polymer solution

An aqueous polymer solution was prepared by carefully adding weighed dry, powdered polymer sample to the solvent taken in a conical flask and gently stirring with a magnetic stirrer for 1 h.

Adsorption experiments

An accurately weighed amount of adsorbent was charged to a 250-mL conical flask. A 20-mL sample of polymer solution was pipetted into the flask, which was corked securely. The mixture was then shaken continuously for 5 h. The system was kept undisturbed for 20 h at the specified temperature and pH. Unless mentioned specifically, the temperature and pH were 25°C and 6.5, respectively. After this period, a portion of the clear solution was withdrawn for determination of polymer concentration.

Both viscometric and turbidimetric methods can be followed to determine an unknown polymer concentration in solution.¹⁶ For determining the concentra-

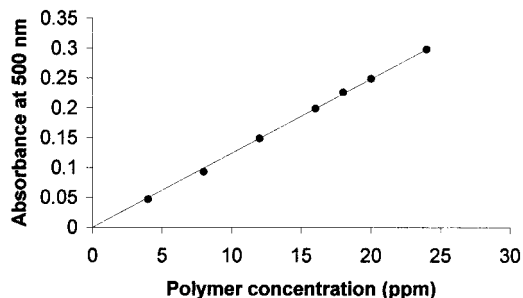


Figure 1 Turbidimetric calibration plot for sample 3.

tion of the anionic polymers used in this work, the method described by Allison et al.¹⁷ was found quite suitable. In this method, anionic polymer in solution was allowed to form a complex with the cationic surfactant Hyamine-1622. The complex made the solution turbid. The turbidity increased with time and attained a constant value after a certain interval of time. Comparison of turbidity at a certain instant after adding the reagent with previously calibrated values gives the unknown polymer concentration. Allison et al. used this method successfully for determining the concentration of PHPA. They used Na-citrate solution as buffer as well as masking agent. The proper time for obtaining stable turbidity results for PHPA was found between 30 and 50 min. They found a straight calibration plot of concentration versus absorbance at 500 nm up to a polymer concentration of 20 ppm.

In the present work, a calibration plot was obtained for each ionic sample. Figure 1 shows the calibration plot for sample 3. For calibration, absorbance of solutions containing the exact amount of polymer, 1% Na-citrate, and 0.8% Hyamine-1622 were determined at a pH of 6.5 in each case. The absorbance at 500 nm was noted after a certain interval of time after adding the reagent. The appropriate time interval for obtaining a stable reading of turbidity was determined separately for each sample. After adding Hyamine-1622 to a polymer solution in 1% Na-citrate aqueous solvent, the absorbance at 500 nm was recorded periodically. It was observed that the absorbance first increased rapidly, after which it attained a constant value. The corresponding time for attaining this constant value was 45 min for sample 3 (Fig. 2). All the calibration plots remained straight at least up to a concentration of 25 ppm.

To determine the time required to attain the adsorption equilibrium, 5 g sand was gently shaken in a 250-mL corked conical flask with 20 mL polymer solution. Aliquots of 0.1 mL from this solution were withdrawn at certain time intervals. Absorbance at 500 nm at a constant dilution was determined each time as described above. Figure 3 shows the increase in adsorption with time of contact for sample 3. For all samples it was observed that the adsorption becomes

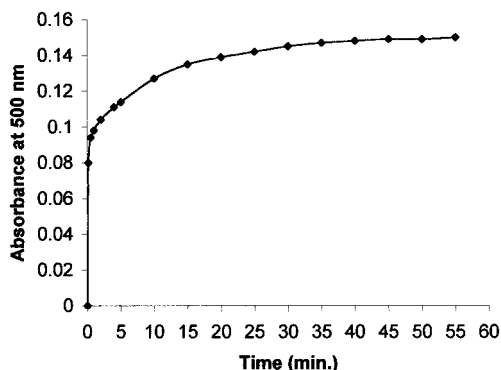


Figure 2 Change of absorbance with time after addition of Hyamine-1622 to 12 ppm sample 3 solution.

almost constant within a period of 4 h. It was also observed that this period is less than 3.5 h for sample 3 adsorbed on bentonite and limestone.

Because AM-NVP is a nonionic polymer, it does not form a complex with Hyamine-1622. Therefore, the viscometric method was used for determining the concentration of this polymer. Because of its nonionic character, there would be little effect of ionic impurities or pH on the solution viscosity of this polymer. Therefore, the viscometric method should yield satisfactory results. In this method, also, a calibration line was obtained by plotting the polymer concentration versus the flow time for polymer solutions of known concentrations through a capillary viscometer. It was observed experimentally that sample 7 gives a straight line at least up to a concentration of 0.1% (Fig. 4). The concentration of polymer solution after adsorption was determined by comparing the flow time of the solution through the same viscometer with the calibration plot.

RESULTS AND DISCUSSION

Adsorption from fresh water

Adsorption isotherms of sample 3 for sand, bentonite, and limestone as adsorbents and fresh water as sol-

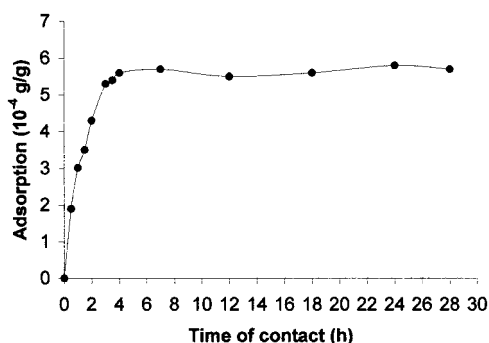


Figure 3 Change of adsorption with increasing time of contact for sample 3.

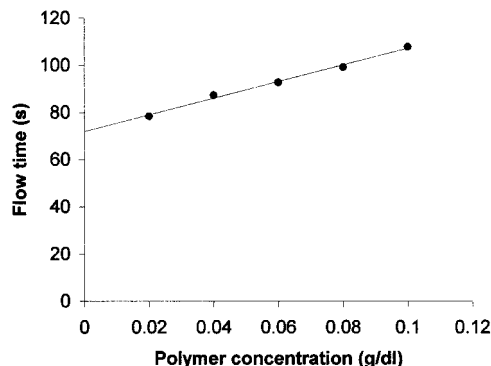


Figure 4 Viscometric calibration curve for sample 7.

vent are presented in Figure 5. It is apparent from the figure that adsorption of the polymer follows the order: bentonite \gg limestone $>$ sand. Adsorption of the polymer on sand is very low in fresh water. This may be attributed directly to the repulsive interaction between the negatively charged sand surface and the anionic polymer in solution. It was observed that adsorption on sand increased slowly to a certain polymer concentration and then attained a constant value. The amount adsorbed remained constant up to an equilibrium polymer concentration of about 0.17%. This corresponds to an initial polymer concentration of 0.2%. Above this concentration, the adsorption increased suddenly to higher values. For polymer solution, the adsorption characteristics are better studied below the concentration of 0.2%, given that above this concentration multilayer adsorption makes it complex.¹⁸ In the present case also the unexpectedly high adsorption in higher polymer concentration may be explained by the multilayer adsorption. From the thermodynamic perspective, this type of isotherm is formed because of multilayer adsorption and the enthalpy of adsorption for the first layer is smaller than that for the subsequent layers.¹⁹ After the completion of monolayer adsorption, further adsorption occurs on the adsorbed polymer layer instead of the sand surface. Probably

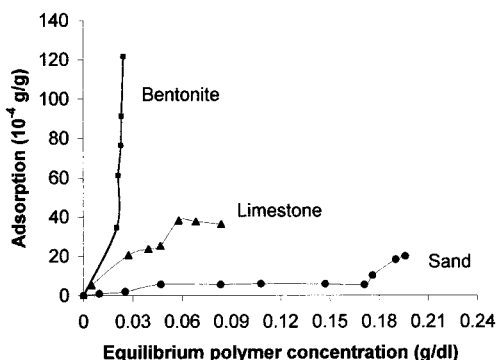


Figure 5 Adsorption isotherms for sample 3 using different adsorbents.

the factors responsible for increasing the adsorption abruptly are (i) lower negative charge density on the adsorbed polymer layer than that on the sand surface and (ii) increased chance of physisorption attributed to entanglement of polymers in solution and on the adsorbed layer.^{20,21}

The higher adsorption on limestone and bentonite can be attributed to their large surface areas. Limestone used for this work contains all the sizes below BS-72 mesh size (212 μm). Consequently, it will have a larger surface area than that of the sand. The particle size of bentonite is extremely small compared to that of the sand used and hence has a much higher surface area.²² The shape of the isotherm for adsorption on limestone stands for multilayer adsorption as well as condensation in pores.¹⁹ In this case also, the enthalpy of adsorption for the first layer is smaller than that for the subsequent layers. Adsorption on bentonite gave an isotherm that stands for multilayer adsorption having lower enthalpy of adsorption for the first layer than that for the subsequent layers.¹⁹ That is why the adsorption increased so abruptly within a very small range of equilibrium polymer concentration.

From the above discussion it may be understood that the nature of adsorption of the polymer on the three adsorbents taken are different in nature. Because the polymer and the experimental conditions are identical, this difference can be attributed to the difference in structure, composition, and surface area of the three adsorbents. However, the present study emphasizes the adsorption behavior on sand surface only. The other adsorbents were taken only for a preliminary understanding of the adsorption behavior of the polymer.

Adsorption from brine

The increase in adsorption of anionic polymer on sand surface on the addition of electrolytes such as NaCl, CaCl_2 , and so forth is a well-established phenomenon.^{10,23,24} The fact is attributed to (i) reduction of electrostatic repulsion between polyelectrolyte and sand surface by charge screening, (ii) specific interac-

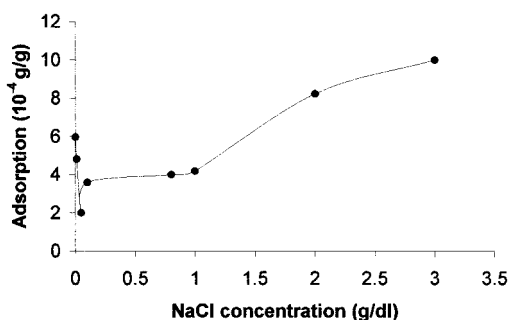


Figure 6 Effect of NaCl on adsorption of sample 3 on sand.

TABLE III
Adsorption of Three Fractions of Sample 3 on Sand

Sample	Intrinsic viscosity (dL/g) at 25°C in 0.1M NaCl	Adsorption ($\times 10^4$ g/g)
1	3.72	4.689
2	3.95	4.750
3	4.44	4.974

tion of cations with polymer decreasing its charge and affinity for the solvent, and (iii) fixation of cations on adsorbent surface reducing surface charge and creating new adsorption sites for the polymer. For sample 3, a similar effect was observed above 1.5% NaCl concentration (Fig. 6); however, the same was not true below this concentration. It is apparent from the figure that the adsorption decreases to a minimum at an NaCl concentration of 0.05% and then increases with further addition of the salt. The behavior of the polymer in higher concentration range is in line with the accepted view.

The adsorption of the polymer on sand surface is physical in nature, which is explained in a later part of this work. Therefore, the amount adsorbed should be proportional to the size of the polymer molecule. This is supported by the qualitative experimental observation that the polymer fraction with higher intrinsic viscosity is adsorbed to a greater degree on the sand (Table III). On the addition of a salt to polymer solution, the salt favors the adsorption by bringing the three changes to the system as mentioned above and at the same time it disfavors the adsorption by allowing the polyanion to coil up by decreasing the intraionic repulsion (i.e., by reducing the effective size). At very low concentration of the salt, probably the cations are stabilized more by the polyanion in solution than by the sand surface. As a result, the cations would prefer to exist in solution than to be adsorbed on the sand surface. Therefore the effect of polymer size reduction is predominant in the very dilute range

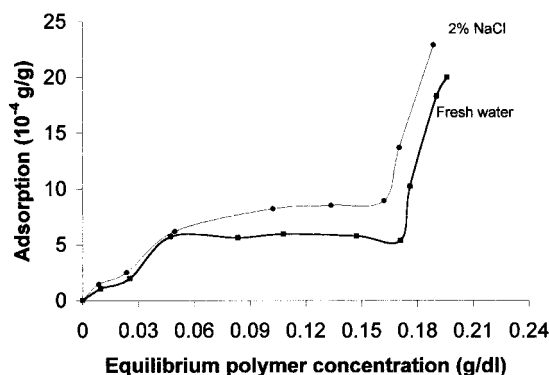


Figure 7 Adsorption isotherm of sample 3 in 2% NaCl and fresh water.

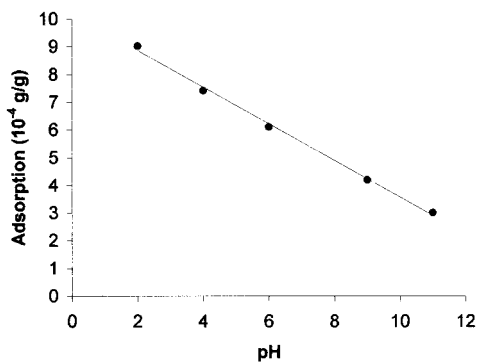


Figure 8 Effect of pH on adsorption of sample 3.

of NaCl concentration. Above the adsorption minimum, the normal salt effects are predominant, increasing adsorption with increasing salt concentration. Chatellier et al.²⁵ also mentioned a damped oscillation profile at the low brine concentration range.

At sufficiently high brine concentration, the shape of the adsorption isotherm is identical to that in fresh water, the only difference being the higher amount adsorbed for the former. Figure 7 shows the adsorption isotherm in 2% NaCl brine. For convenience, the isotherm without brine is also presented in the same figure.

Effect of pH on adsorption

Figure 8 shows the effect of pH on adsorption of sample 3 on the sand surface. The plot of pH versus adsorption is a straight line with a negative slope. On decreasing the pH the [H⁺] in the solution increases. The negative charge density on the sand surface is partially compensated by the accumulation of H⁺ on the surface; thus the adsorption increased with decreasing pH. Again on increasing the pH, the surface became increasingly negative because of the accumulation of OH⁻ groups, which reduced the adsorption.²⁶

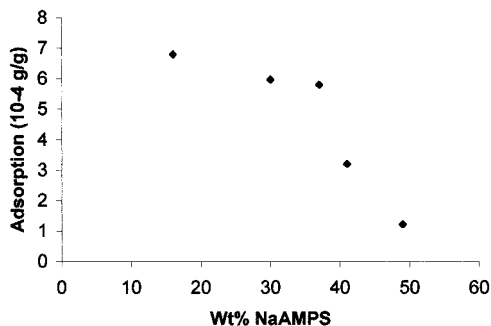


Figure 9 Effect of wt % ionic comonomer on adsorption.

TABLE IV
Adsorption Data of Different Types of Polymer in Identical Conditions

Copolymer	Adsorption ($\times 10^4$ g/g)
Sample 3	5.96
Sample 6	4.32
Sample 7	11.41

Effect of percentage ionic character of copolymer on adsorption

Because the sand surface is negatively charged, the adsorption is expected to decrease with increasing the fraction of anionic group in the copolymer, which was confirmed experimentally. Figure 9 shows the adsorption of sample 1 to sample 5 on sand. It is apparent from the figure that the decrease in adsorption is abrupt from 37 wt % NaAMPS onward and the adsorption is negligible when the ionic monomer reaches 50 wt %.

It may be seen from Table IV that a nonionic polymer of comparable intrinsic viscosity is adsorbed more than an anionic one. Sample 7 (i.e., AM-NVP) is a nonionic polymer that is adsorbed 11.41×10^{-4} g/g, whereas 5.96×10^{-4} g/g of sample 3 and 4.32×10^{-4} g/g of sample 6 are adsorbed under the same experimental conditions. The smaller value for sample 6 may be understood to be attributed to the higher mol % of anionic group present in the copolymer than that in sample 3. Even in the presence of 3% NaCl, the adsorption of sample 3 is lower than that of sample 7.

Thermodynamics of adsorption

Details of thermodynamics were not studied in this work. The effect of temperature on adsorption behavior of the polymer solution was studied to understand the nature of adsorption. The principal test for distinguishing chemisorption from physisorption used to be the enthalpy of adsorption.²⁷ Values of enthalpy of adsorption less negative than -25 kJ mol⁻¹ are taken to signify physisorption and values more negative than -40 kJ mol⁻¹ are taken to signify chemisorption.

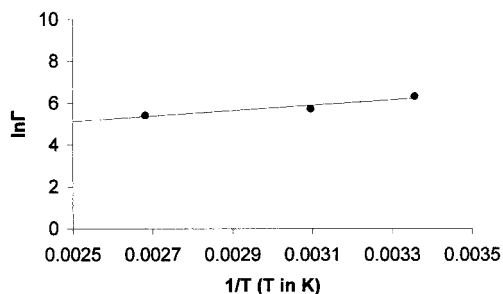


Figure 10 Plot of ln Γ versus 1/T (T in Kelvin).

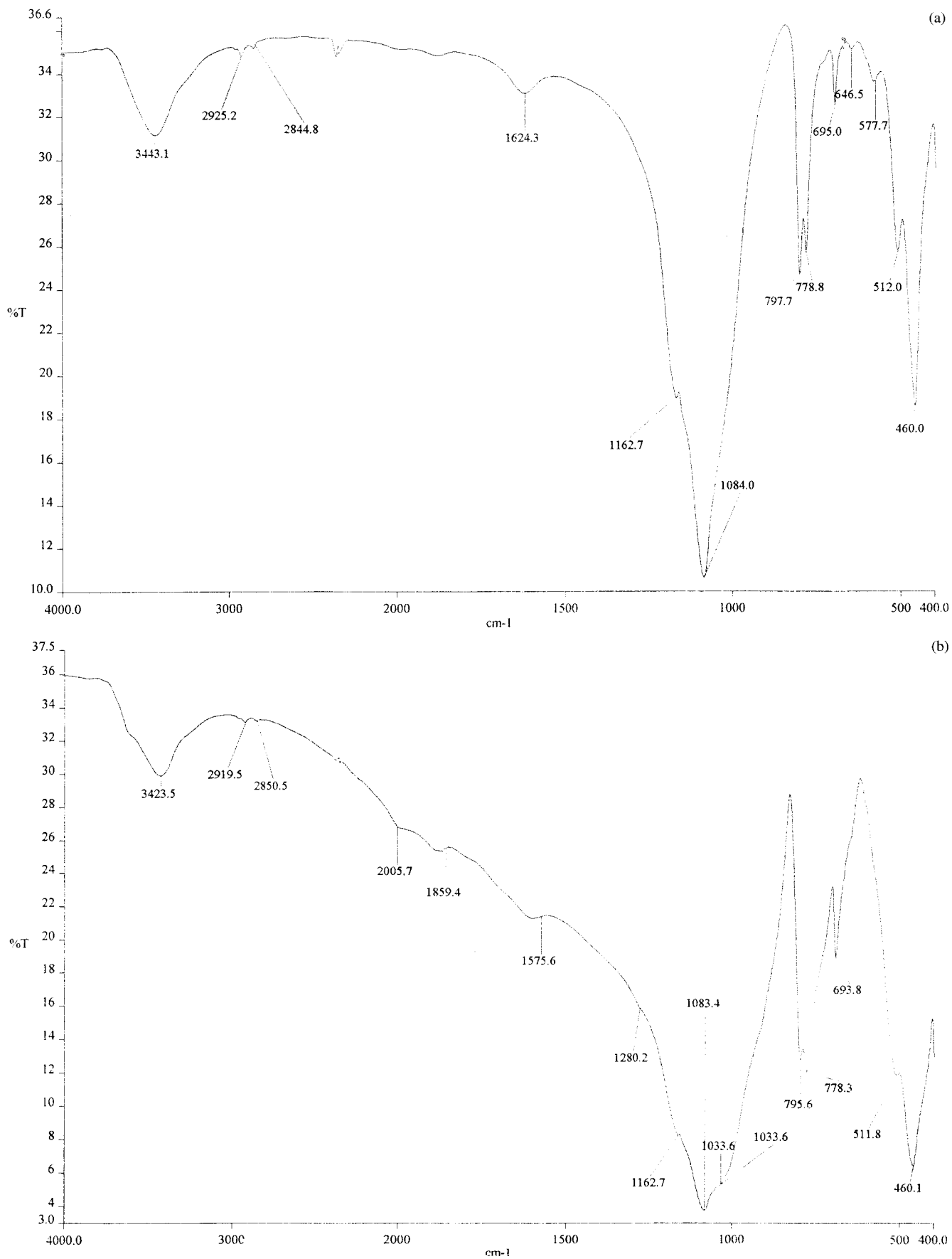


Figure 11 (a) IR spectra of sand before adsorption. (b) IR spectra of sand after adsorption. (c) IR spectra of the polymer sample 3.

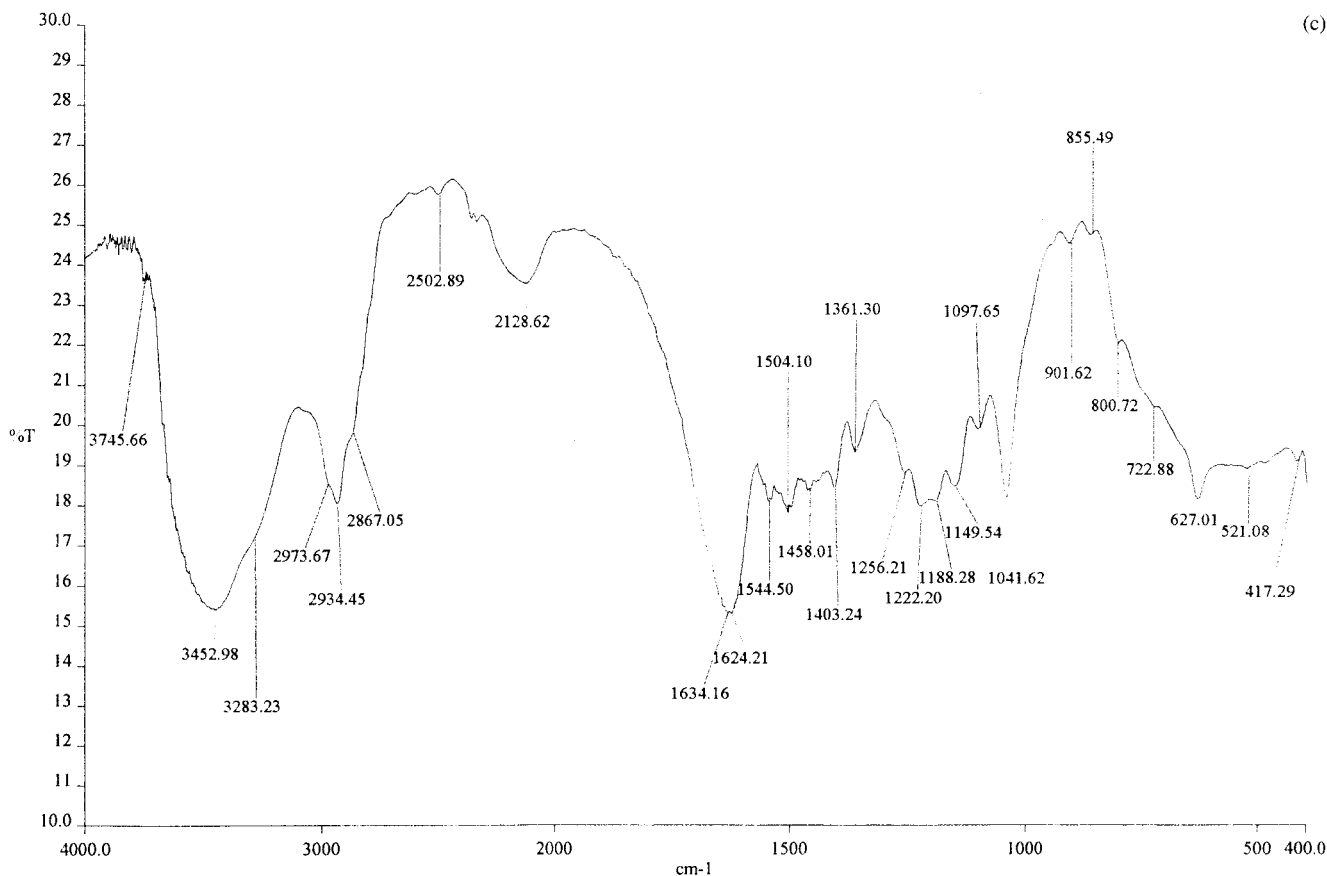


Figure 11 (Continued from the previous page)

However, this criterion is by no means foolproof. Still the enthalpies are widely used to establish the nature of adsorption.

The Clausius–Clapeyron equation can be used to evaluate the enthalpy of adsorption,²⁸ expressed as

$$\Delta H_{\text{ads}} = -R[d \ln \Gamma / d \ln(1/T)]_0 \quad (1)$$

where ΔH_{ads} is the enthalpy of adsorption and Γ is the amount adsorbed in $\mu\text{g/g}$ at temperature T in absolute scale at surface coverage θ . A plot of $\ln \Gamma$ versus $1/T$ will give the value of ΔH_{ads} from the relation

$$\text{Slope} = -(1/R)\Delta H_{\text{ads}} \quad (2)$$

Figure 10 shows the experimental plot for sample 3, which yields a ΔH_{ads} value of $-10.8 \text{ kJ mol}^{-1}$. This is typical of physisorption. Physical adsorption is also supported by the IR spectra of the sand before and after adsorption [Fig. 11(a), (b)]. For convenience, spectra of the polymer are also presented in Figure 11(c). It is apparent from the figures that no significant change in the sharp sand peaks occurs after adsorption. The widening of the peak at 1084 cm^{-1} can be attributed to the overlapping of the sand peak with the

small polymer peaks occurring at the same region. Occurrence of some small new peaks in the $1000\text{--}2000 \text{ cm}^{-1}$ region as well as the fingerprint region and disappearance of the peak at 1624 cm^{-1} in the spectra of sand after adsorption can also be explained by considering the overlapping of peaks. However, the shifting of the peak at 3452 cm^{-1} of the pure polymer to 3423.5 cm^{-1} in the adsorbed sand spectra may indicate the formation of hydrogen bonding between the silica surface (i.e., the oxygen atom) and the secondary amide group of the NaAMPS monomer.

CONCLUSIONS

The NNDAM–NaAMPS copolymer is suitable for EOR application at high-temperature reservoirs at least around 120°C and having high-density brine fluid. It is observed from its adsorption characteristics that the adsorption is not so high compared to that of nonionic polymers or PHPA used in EOR. At neutral or basic pH the adsorption is very low. Also at high temperature the adsorption is low. A sufficient anionic content helps to reduce adsorption. Thus it can be concluded that the NNDAM–NaAMPS copolymer can be used without economic prohibition at high-temper-

ature reservoirs, at least for the sandstone reservoirs for EOR.

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