Characterization of Acrylamide Polymers for Enhanced Oil Recovery

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ABSTRACT: Four different copolymers, of acrylamide and acrylic acid, acrylamide and 2-acrylamido-2-methyl propane sulfonic acid, N,N-dimethylacrylamide and acrylic acid, and N,N-dimethylacrylamide and 2-acrylamido-2-methyl propane sulfonic acid (sodium salts), were prepared. The copolymers were characterized by their intrinsic viscosities and monomer ratios and with IR, ^{1}H-NMR, and X-ray diffraction (XRD) spectroscopy. No crystallinity was observed by differential thermal analysis, and this was well supported by XRD. All the polymers showed low decomposition temperatures. A number of decomposition temperatures were observed in differential thermogravimetry thermograms because of the elimination of gases such as CO_2, SO_2, CO, and NH_3. The replacement of the acrylate group with a sulfonate group produced polymers that were more compatible with brine, whereas the replacement of acrylamide with a more hydrophobic group such as N,N-dimethylacrylamide produced a more shear-resistant polymer. A N,N-dimethylacrylamide-co-sodium-2-acrylamido-2-methyl propane sulfonate copolymer was better with respect to thermal stability when the polymer solution was aged at 120°C for a period of 1 month. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 87: 1869–1878, 2003

INTRODUCTION

With increasing worldwide demands on the energy sector, polymers have gained immense importance as enhanced oil recovery agents among oilfield chemists.[1–3] Enhanced oil recovery is going to play a crucial role in the production of petroleum, which is so vital for the development of any country. Although the consumption of crude oil has increased sharply in recent years, there is a decline in oil reservoirs. In this context, designing a suitable agent for the tertiary recovery of oil from high-temperature reservoirs with high-density brine fluid is considered a challenging job for chemists. Although polyacrylamide, especially partially hydrolyzed polyacrylamide, is considered the best mobility control agent in surfactant–polymer and polymer-augmented water flooding processes, it is not suitable for high-temperature reservoirs (>90°C) with high-density brine fluids.[4–6] The ideal candidate for the enhanced recovery of oil from such reservoirs should have long-term thermal stability under the reservoir conditions of temperature and salinity.[7] Moreover, it should be cost-effective.

Substituted polyacrylamides, or polyacrylamides copolymerized with a suitable monomer, can yield a better product that may be thermally stable at least for a period. Polyacrylamide is not thermally stable be-cause the —NH_2 group is hydrolyzed at 80–90°C: this leads to the formation of a solution of very low viscosity under reservoir conditions.[8] If the hydrogens of the amide group are replaced by methyl groups, this effect should practically be eliminated. Moreover, polymers containing —SO_3^- groups are expected to offer higher stability in solution, on account of their stronger hydrogen bonding, than polymers containing —CO_2^- groups. Therefore, sodium-2-acrylamido-2-methyl propane sulfonate (AMPS) should be more suitable than sodium acrylate (AA) as a comonomer with substituted acrylamide (AM). Also, —SO_3^- should help with the water solubility of the polymer more than —CO_2^- because of the former's stronger hydrophilicity.

From a rheological point of view, the presence of some hydrophobic groups such as —CH_3, along with hydrophilic groups, may help with shear resistance.[9] Again, the presence of bulky side chains should assist in the retention of a comparatively higher solution viscosity after the reduction of the solution viscosity by salt than that obtained with smaller side chains.

Keeping in mind all these points, we selected and prepared in our laboratory four different copolymers of AM or substituted AM. The copolymers were characterized by potentiometry, IR, ^{1}H-NMR, X-ray diffraction (XRD), thermogravimetry, and intrinsic viscosities. The copolymers were poly(acrylamide-co-sodium acrylate) (AM–AA), poly(acrylamide-co-sodium-2-acrylamido-2-methyl propane sulfonate) (AM–AMPS), poly(N,N-dimethylacrylamide-co-sodium acrylate) (NNDAM–AA),

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TABLE I
Synthesis, Composition, and Intrinsic Viscosities of Copolymers

Sample	Comonomer used A	Comonomer used B	Comonomer in reaction mixture (wt %) A	Comonomer in reaction mixture (wt %) B	Comonomer in copolymer (wt %) From ^{1}H-NMR A	Comonomer in copolymer (wt %) From ^{1}H-NMR B	Comonomer in copolymer (wt %) From potentiometry A	Comonomer in copolymer (wt %) From potentiometry B	Intrinsic viscosity in 0.1M NaCl at 27°C (dL/g)
1	AM	AA	70	30	60.2[a]	39.8[a]	64.4	35.6	14.4
2	AM	AMPS	70	30	60.3	39.7	66.6	33.4	5.51
3	NNDAM	AA	70	30	62.0	38.0	67.8	32.2	5.44
4	NNDAM	AMPS	70	30	57.9	42.1	62.8	37.2	4.75

[a] Value obtained by correction of the potentiometric data for the gravimetric error (due to the presence of moisture), determined from TGA.

and poly(N,N-dimethylacrylamide-co-sodium-2-acrylamido-2-methyl propane sulfonate) (NNDAM–AMPS).

EXPERIMENTAL

Materials

Copolymers of AM and N,N-dimethylacrylamide (NNDAM) and of acrylic acid (sodium salt) and AMPS were prepared in the laboratory. AM, obtained from Merck–Schuchardt (Germany), was recrystallized from a water–ethanol mixture. NNDAM, obtained from Aldrich Chemical Co., was exposed over calcium hydride for about 24 h for the removal of stabilizers and was then purified by distillation *in vacuo*. Purified acrylic acid purchased from G. S. Chemicals (New Delhi, India) was used as received. AMPS, obtained from Merck–Schuchardt, was purified by recrystallization. Ammonium persulfate and sodium metabisulfite were obtained from CDH (New Delhi, India) and purified by recrystallization.

Preparation of the polymers

The synthesis of AM copolymers is a well-established phenomenon.[10] Distilled or purified monomers were dissolved in degassed and distilled water in the ratio given in Table I to obtain a 12 wt % aqueous solution. Residual oxygen was removed by nitrogen being bubbled through the solution for an hour under constant stirring at 25°C. To this solution, recrystallized ammonium persulfate (0.15 wt % monomer) and sodium metabisulfite (0.2 wt % monomer) solutions were added slowly. The reaction was then allowed to occur at 25°C for 6 h. The final solution was clear and highly viscous. The solution was diluted to 2–3 times its volume. A part of the polymer was then isolated by precipitation with acetone and dried *in vacuo* for 10–15 h.

The acid content in the copolymer was then determined by the potentiometric titration method. The remaining part of the reaction mixture was neutralized with NaOH (2% solution). The final copolymer was isolated by precipitation with acetone and dried in a vacuum oven for 10–15 h.

Instrumentation

Kinematic viscosity is measured with an Ubbelohde viscometer (fit with an automatic Schott Gerate AVS 400 instrument, Germany). Solution viscosity is measured with the help of a Brookfield LVTDV II viscometer equipped with an Ultra Low (UL) adapter (Stoughton, MA). A rotoviscometer having a maximum shear rate of 1312 s^{-1} is also used in some cases. Thermogravimetric experiments are performed in a Universal V2.5H TA instrument (New Castle, DE) in a nitrogen atmosphere at a heating rate of 8°C/min. IR spectra are taken with a PerkinElmer IR 883 instrument (Beaconsfield, UK), taking samples in KBr pellets. ^{1}H-NMR spectra are taken with a Bruker 300-MHz instrument (Fallanden, Switzerland) using TMS as the reference and D$_2$O as the reference and D$_2$O as the solvent. Wide-angle XRD are taken in an X-ray diffractometer, Type JDX-11P3A (JEOL, Tokyo, Japan).

RESULTS AND DISCUSSION

The intrinsic viscosities of the copolymers and the ratios of the monomers in the copolymers are presented in Table I. ^{1}H-NMR and potentiometric titrations were used to determine the monomer ratios in the copolymers (Table I). The differences between the two results could be rationalized by a consideration of the moisture contents of the samples (obtained from thermogravimetry data shown later in Table IV). The ^{1}H-NMR and IR data of the samples are presented in Tables II and Table III, respectively. As expected, both the ^{1}H-NMR and IR spectra confirmed the presence of different monomers in the copolymers. A typical presentation of the ^{1}H-NMR and IR spectra of one of the copolymers is shown in Figure 1(a,b).

TABLE II
^{1}H-NMR Data and Interpretation

Sample 1		Sample 2		Sample 3		Sample 4	
δ	Interpretation	δ	Interpretation	δ	Interpretation	δ	Interpretation
1.52	—CH$_2$—	1.33	—C—CH$_3$ with CH$_3$	1.61	—CH$_2$—	1.59	—C—CH$_3$ with CH$_3$
2.08	—CH— with C=O	1.53	—CH$_2$—	2.17	—CH—	1.69	—CH$_2$—
		1.70	—CH$_2$—SO$_3$—	2.88 2.94	—N with CH$_3$ and CH$_3$	2.27	—CH—
		2.27	—CH—			3.01 3.08 3.19	—N with CH$_3$ and CH$_3$

Thermogravimetric analysis (TGA)

The application of thermogravimetric tools such as differential thermal analysis (DTA), TGA, and differential thermogravimetry (DTG) is very significant in characterizing a polymer.[11–14] The data in Table IV present the thermogravimetry (DTA, TGA, and DTG) of all four polymers. In the DTA thermograms, no detectable crystallinity was observed in any of the four samples. This observation was also supported by the XRD spectra of the samples, indicating no crystallinity. All the polymer samples had low decomposition temperatures. The glass-transition temperatures (T_g's) of the samples were determined from the DTA thermograms.[15] As presented in Table IV, T_g of the

NNDAM–AA copolymer was not detectable from DTA [Fig. 1(c)]. There was no indication of a small, sudden endothermic change in the DTA thermogram of the polymer.

T_g of a copolymer depends on several factors, physical and chemical.[14,16–18] Previously, it was thought to depend on the physical properties of the homopolymers composed of the comonomers of the copolymer.[19] The properties include the specific volume, molecular cohesive energy, and chain stiffness. Considering these properties, Fox proposed the following equation:[19]

$$1/T_{gC} = W_A/T_{gA} + W_B/T_{gB}$$

TABLE III
IR Data and Interpretation

Sample 1		Sample 2		Sample 3		Sample 4	
Peak (cm^{-1})	Interpretation	Peak (cm^{-1})	Interpretation	Peak (cm^{-1})	Interpretation	Peak (cm^{-1})	Interpretation
3420 (b)	N–H and O–H str.	3440 (b)	N–H and O–H str	3416 (b)	O–H str.	3440 (b)	N–H and O–H str.
2930 (w)	C–H str.	2925 (w)	C–H str.	2870 (w)	C–H str.	2944 (w)	C–H str.
1625 (s)	C=O str. of 1° amide	1678 (s)	C=O str. of 2° amide	1628 (s)	C=O str. of 3° amide	1638 (s)	C=O str. of 3° amide
1596 (s)	O–C–O$^-$ str.	1647 (s)	C=O str. of 1° amide	1592 (s)	O–C–O= str.	1610 (s)	C=O str. of 2° amide
1377 (m)	–CH$_2$– ben.	1417 (m)	–C–H ben. of –CH$_2$–	1388 (m)	C–H ben. of gem-dimethyl	1380 (m)	C–H ben. of gem-dimethyl
1342 (m)	–C–H ben.	1385 (m)	C–H ben. of gem-dimethyl	1354 (m)	C–H ben. of –CH$_2$–	1348 (m)	C–H ben. of –CH$_2$–
		1065 (w)	–SO$_3$$^-$			1175 (w)	
		1140 (w)				1034 (m)	–SO$_3$$^-$

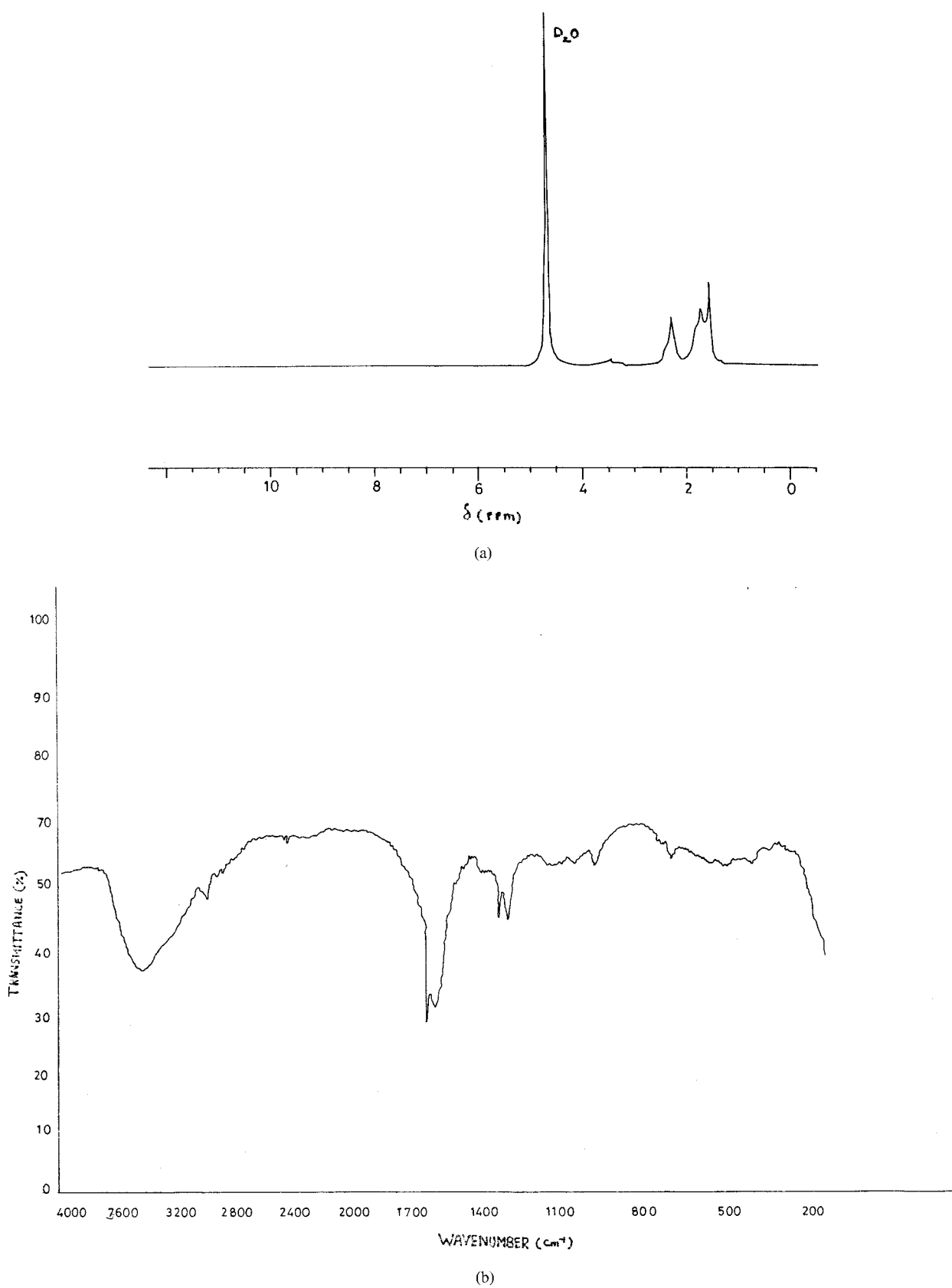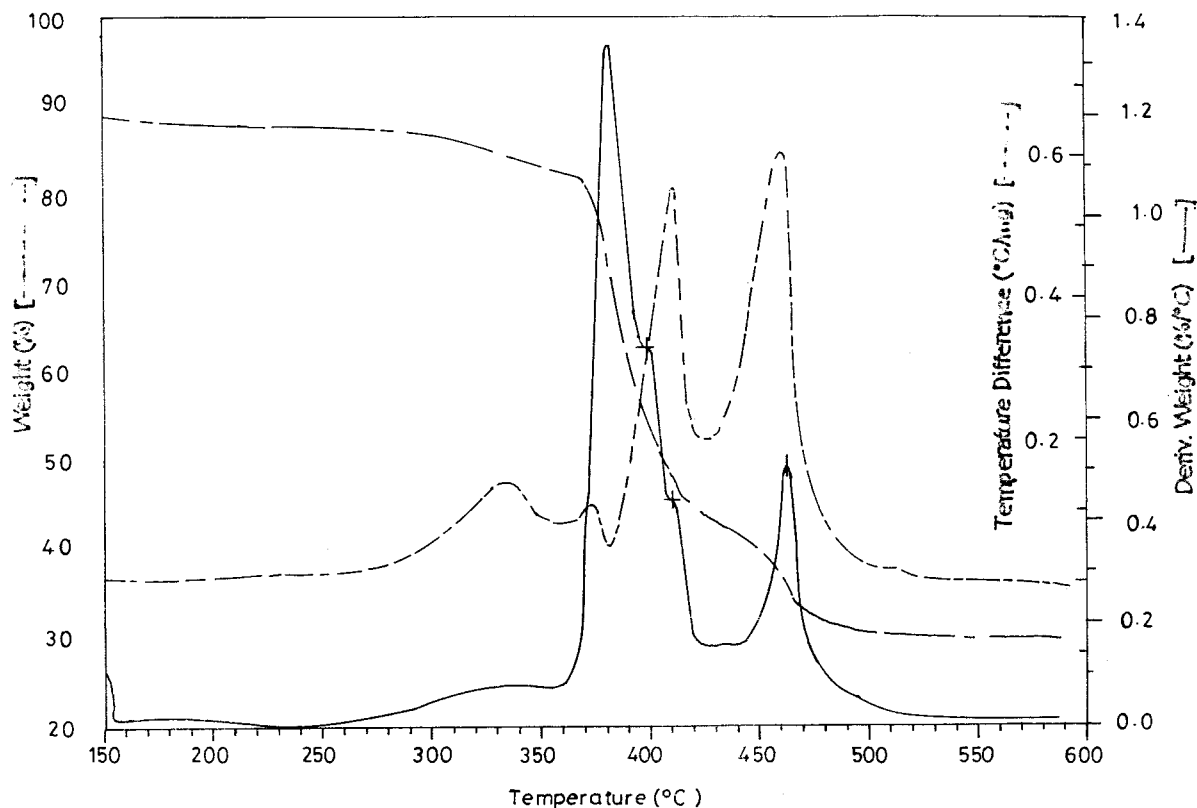str: stretching vibration, ben: bending vibration.

(a)

(b)

Figure 1   (a) ^{1}H-NMR spectra of polymer 2; (b) IR spectra of polymer 2; and (c) DTA, TGA, and DTG thermograms of polymer 3.

(c)

Figure 1 (*Continued from the previous page*)

where T_{gC}, T_{gA}, and T_{gB} are the glass-transition temperatures of copolymer C and homopolymers A and B (which comprise C), respectively, and W_A and W_B are the weight fractions of comonomers A and B in copolymer C. With this equation, the T_g values of all four copolymers (Table IV) were calculated. The deviation from the experimental values can be rationalized by a consideration of the chemical effects of the functional groups of the comonomers. For copolymers, Johnston[20] proposed a modified equation relating a copolymer's T_g to its composition. However, this is out of the scope of this work and, therefore, is not discussed.

Table IV shows that polymers 1 and 4 had comparable T_g's, whereas polymer 2 had a lower T_g. However, these were random copolymers with different monomer compositions. Therefore, it was extremely difficult to explain their secondary phase-transition behavior, such as the glass transition, which depended on several chemical and physical factors. Still, T_g data can indicate the most ordered and stiffest arrangement of macromolecules in sample 4.

All four copolymers showed more than one decomposition temperature in DTG and DTA thermograms.[21] With increasing temperature, there was breakage of the bonds in the copolymers, resulting in

TABLE IV
DTA, TGA, and DTG Data

Sample	Moisture content (wt %)	T_g (°C) From DTA	T_g (°C) From Fox equation[a]	Decomposition temperature (°C) from DTG peak (with % weight lost) T_1	T_2	T_3	T_4	T_5
1	12	235	186	246 (8%)	320 (6%)	358 (6%)	369 (7%)	390 (10%)
2	12	205	116	280 (10%)	310 (11%)	376 (24%)	459 (15%)	544 (13%)
3	11	—	116	336 (4%)	381 (24%)	398 (11%)	410 (9%)	463 (6%)
4	8	239	85	238 (3%)	275 (4%)	316 (9%)	360 (24%)	522 (20%)

[a] The T_g values of polyamide, poly(sodium acrylate, and poly(N,N-dimethylacrylamide) were obtained from ref. 25 whereas that for poly(sodium-2-acrylamide-2-methylpropane sufonate) was determined from DTA.
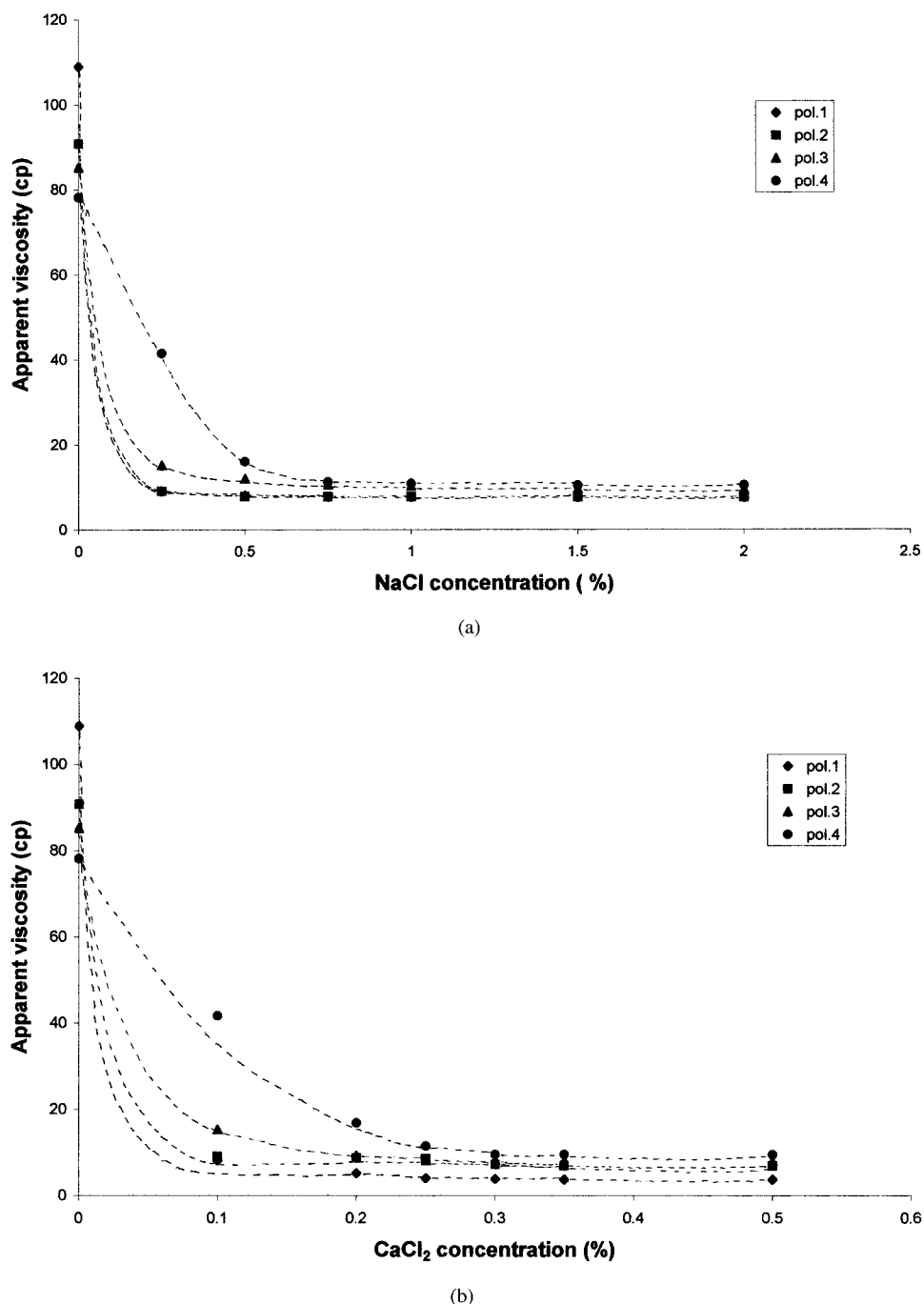
(a)

(b)

Figure 2 (a) NaCl, (b) $CaCl_2$, and (c) $MgCl_2 \cdot 4H_2O$ concentration dependence of the aqueous solution viscosity of a 0.2% solution of the polymers at 7.32 s^{-1} and 25°C.

the elimination of different types of gases, such as CO_2, SO_2, CO, and NH_3. Five different decomposition temperatures, indicated by DTG peaks along with the weight percentage lost, were considered for the evaluation of the thermal behavior of the copolymers (Table IV). Before proceeding to a higher temperature in thermogravimetric experiments, each sample was heated to 150°C at a uniform heating rate and kept there isothermally for 5 min for the complete elimina-

tion of absorbed moisture. The values of the moisture content (Table IV) support the deviation of the monomer composition data determined potentiometrically from those data obtained from ^{1}H-NMR. It can be observed in Table IV that all the decomposition temperatures of AM–AMPS decreased on the replacement of AM with NNDAM. However, all the decomposition temperatures of AM–AA increased on the replacement of AM with NNDAM.
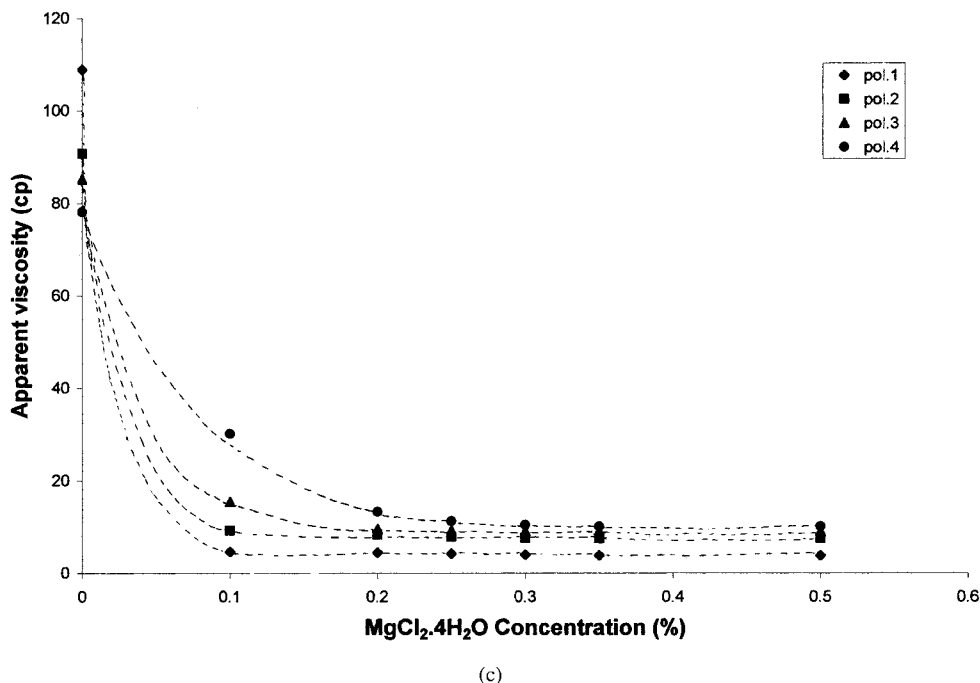
(c)

Figure 2 (*Continued from the previous page*)

Rheological behavior

Effect of the brine concentration on the solution viscosity

It can be observed in Figure 2(a–c) that the effect of Ca^{2+} and Mg^{2+} was much stronger than that of Na^+ in reducing the solution viscosity of all four copolymer solutions. In each case, the viscosity decreased to a certain concentration of added salt and then remained constant, even on the addition of more salts to the solution. This can be explained by a consideration of the polyelectrolyte behavior of the copolymers in brine-free water. With an increasing cation concentration in the solution, the stretched polyelectrolyte chain started shrinking because of a reduction in intra-anionic electrostatic repulsion. At a certain salt concentration, the amount of cations present was sufficient to complete the shrinking, making the copolymer behave like a nonelectrolyte.[22] Therefore, beyond that salt concentration, the addition of salt could not reduce the viscosity more, and this is typical of a nonelectrolyte.
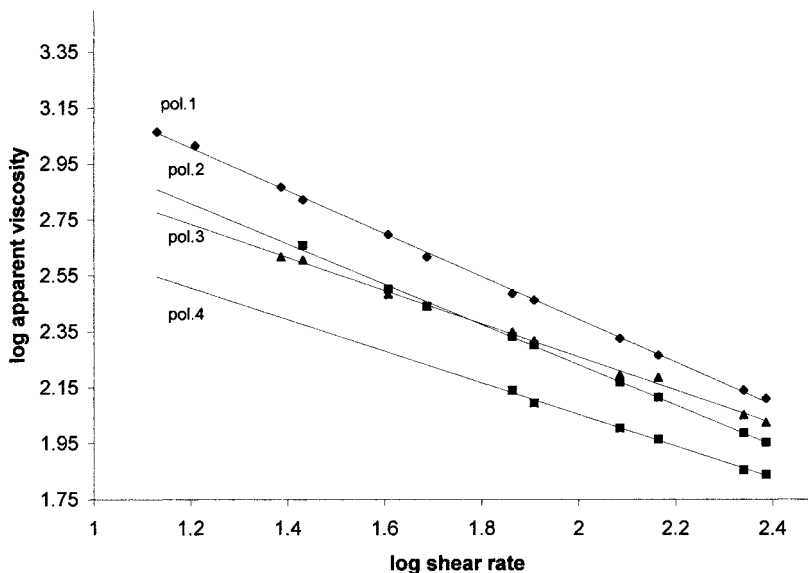

Figure 3 Shear rate dependence of the apparent viscosity of a 0.8% aqueous solution of the polymers at 25°C (logarithmic scale).

TABLE V
Power-Law Constants for Copolymers at 25°C
(Polymer Concentration = 0.8%)

Power-law constant	Sample 1	Sample 2	Sample 3	Sample 4
K (mPa s^n)	8454.73	4704.35	2795.76	1537.45
n	0.2336	0.2795	0.4065	0.4342

Except for polymer 1, the minimum viscosity obtained by the addition of salt was comparable for all three cations: Ca^{2+}, Mg^{2+}, and Na^+. However, for polymer 1, the solution viscosity decreased to a larger extent in the presence of bivalent cations than in the presence of monovalent cations. This can be explained by the fact that polymer 1 formed complexes with the bivalent ions, which were precipitated consequently on further addition. However, no precipitation was observed with the addition of excess bivalent ions in the other three polymer solutions. Because of the replacement of $-CO_2^-$ by $-SO_3^-$ in polymers 2 and 4, the polymer solutions were compatible with bivalent ions. Although $-CO_2^-$ was present in polymer 3, the bulky N,N-dimethyl group was responsible for the compatibility of the polymer solution with bivalent ions.

Effect of the shear on the solution viscosity

It is apparent from Figure 3 that all the copolymer solutions showed non-Newtonian shear-thinning behavior obeying a power law (Table V), at least within the experimental range of the shear rate (1–1312 s^{-1}).

The values of n (<1) supported non-Newtonian thixotropic behavior, whereas the values of K (decreasing from polymer 1 to polymer 4) indicated the higher apparent viscosity of a solution of an identical polymer concentration.[23] It may be mentioned that a higher apparent viscosity in a brine-free aqueous solution of a polyelectrolyte is of no importance if it has a lower relative resistance factor in brine water than another polyelectrolyte of a lower apparent viscosity in brine-free water with a higher relative resistance factor in brine water.[24]

It can observed from Figure 3 that polymer 1 was more shear-sensitive than the other polymers. In fact, polymer 4 was more shear-resistant than the other polymers. The shear resistance of polymer 2 was comparable to that of polymer 1, whereas the shear resistance of polymer 3 was comparable to that of polymer 4. This may support the fact that the presence of hydrophobic groups in polymers 3 and 4 could contribute to the shear resistance of aqueous polymer solutions.

In another experiment, all four polymer solutions were continuously sheared at a constant shear rate for a long period of time. After the withdrawal of shear, the polymer solutions were allowed to stand for a period. All the polymer solutions exhibited time-dependent thixotropic behavior under shear (Fig. 4). Polymers 2–4 partially regained viscosity after aging for some time after the withdrawal of shear. However, the viscosity of polymer 1 decreased further, even after the withdrawal of shear, and never gained viscosity again. When the polymer solution was continuously sheared at a constant shear rate, the intramac-
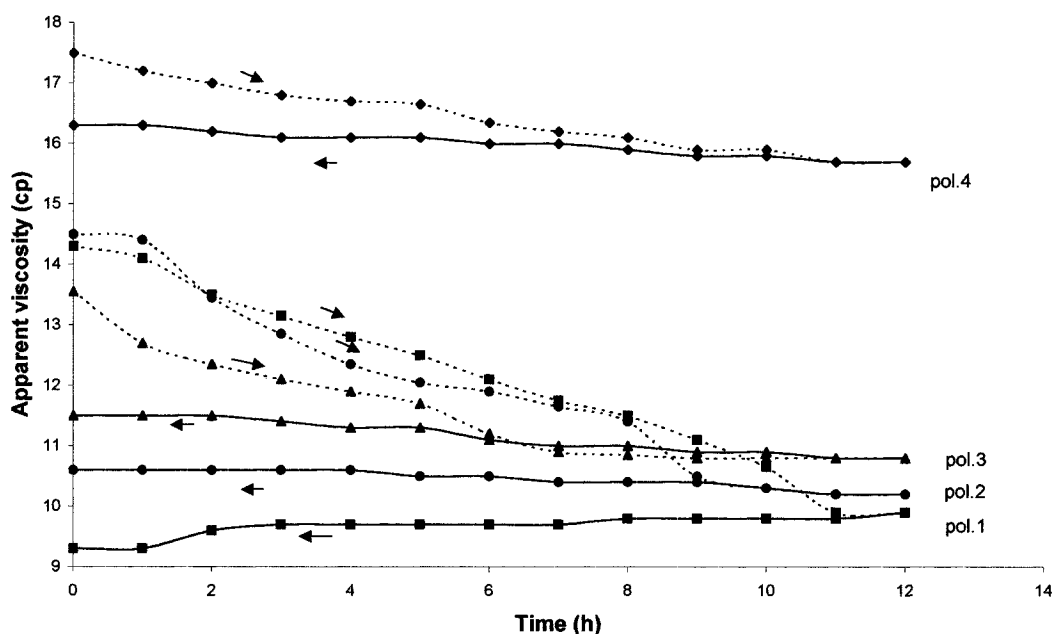

Figure 4 Time-dependent hysteresis loop for a 0.02% aqueous solution under dynamic (dashed lines; constant shear rate = 36.72 s^{-1}) and static (bold lines) conditions at 30°C.
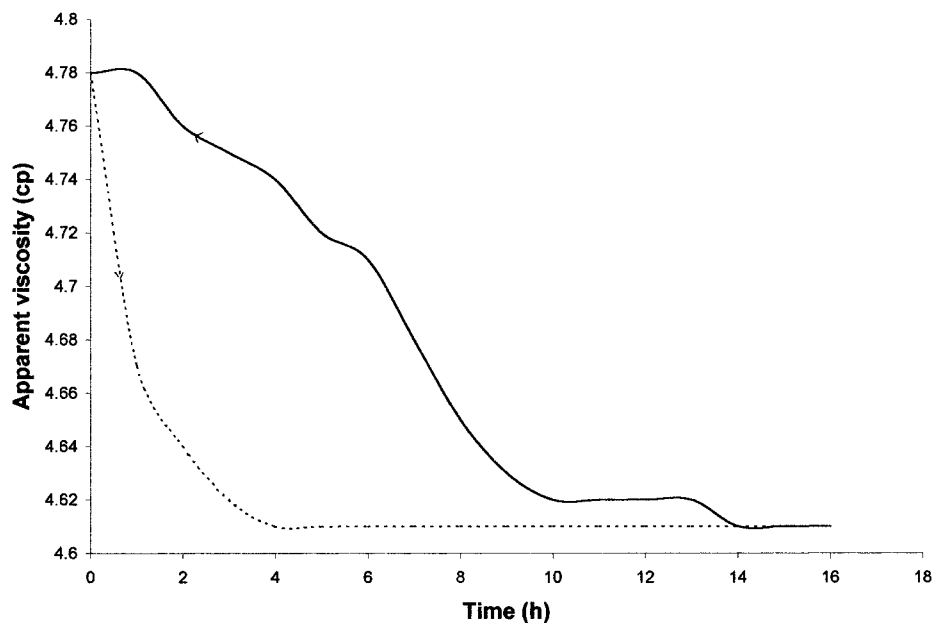
Figure 5 Time-dependent hysteresis loop for polymer 4 (0.02%) in a 0.01% NaCl solution under dynamic (dashed line; constant shear rate = 36.72 s^{-1}) and static (bold line) conditions at 36.72 s^{-1} and 30°C.

roion interaction between the charged ions progressively diminished, showing a reduction in the hydrodynamic volume of the polymer and causing a decrease in the apparent viscosity with time. If the polymer solution was sheared for a long period, the rate of the reformation of the structure of the polymer could increase with time as the possibility of new structural linkages increased.[23] After a certain period, a dynamic equilibrium was reached, beyond which only the equilibrium viscosity was observed. Among polymers 2–4, the amount of viscosity regained was highest in 4, less in 3, and least in 2; this order may also support the contribution of the hydrophilic group to shear resistance. In addition, the stronger hydrogen bonding ability of the —SO$_3^-$ group should have helped with the reformation of the structures in polymers 2 and 4 during shearing, which should have been less in polymers 1 and 3 because they had the —CO$_2^-$ group instead of —SO$_3^-$.

In the presence of brine, the macroions shrank and became more like spheres than a stretched chain. This helped in resisting the deformation of the structure on shearing and, therefore, the reduction in viscosity on shearing. Experimentally, it was verified for polymer 4 (Fig. 5): a slight decrease in viscosity occurred temporarily, but the viscosity was regained fully after the withdrawal of shear.

Effect of high-temperature aging on the solution viscosity

The thermal stability of the polymer solutions were studied by aging at 120°C under a nitrogen atmosphere for a period of 1 month. It can be observed from Table VI that polymer solutions of comparable concentrations differed in their stability. Polymer 1 lost efficacy very fast. The replacement of the AA group of polymer 1 by AMPS in polymer 2 improved the stability slightly but not sufficiently. The same extent of improvement was obtained by the replacement of the other monomer, AM, by NNDAM in polymer 3. This showed that monomers AA and AM were equally responsible for the low thermal stability of the copolymers. For a further improvement in the

TABLE VI
Effect of High-Temperature Aging on Solution Viscosity

Sample no (in 0.8% aqueous solution)	Solution viscosity (cp) at 1312 s^{-1} and 25°C after aging at 120°C for									
	0 days	1 day	4 days	6 days	8 days	10 days	15 days	20 days	25 days	30 days
1	39.2	8.0	7.0	—	—	—	—	—	—	—
2	31.6	12.3	7.5	6.0	—	—	—	—	—	—
3	28.0	15.1	8.0	6.0	—	—	—	—	—	—
4	29.9	26.5	23.9	22.0	21.6	21.0	19.8	19.6	19.4	19.3

thermal stability, monomers AM and AA were replaced by NNDAM and AMPS, respectively, in polymer 4. Moreover, polymer 4 exhibited better thermal stability than the other three polymers. The loss of solution viscosity for this polymer was minimal. About 60% of the solution viscosity was retained even after 1 month of aging at 120°C.

CONCLUSIONS

Four different AM copolymers were studied. No crystallinity was observed by DTA, and this was well supported by XRD. Five decomposition temperatures were observed in DTG thermograms because of the elimination of gases such as CO_2, SO_2, CO, and NH_3. The replacement of the AA group with a sulfonate group produced polymers that were more compatible with brine, whereas the replacement of AM with a hydrophobic group containing a monomer such as NNDAM produced more shear-resistant polymers. Considering all these points, we can say that NNDAM–AMPS is a better copolymer with respect to the brine compatibility, shear resistance, and thermal stability necessary for enhanced oil recovery.

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