

Rheological Behavior of *N,N*-Dimethyl Acrylamide–Acrylamido Methylpropane Sulphonate Copolymer

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ABSTRACT: Copolymer of *N,N*-dimethylacrylamide (NNDAM) and sodium 2-acrylamido-2-methylpropanesulfonate (NaAMPS) have been prepared by free-radical copolymerization and characterized with the help of molecular weight, molecular weight distribution, intrinsic viscosity, and monomer ratio in the copolymer. The solution behavior of a copolymer containing 26.62 wt % NaAMPS is studied in different solvents, namely, water (W), dimethyl sulfoxide (DMSO), ethylene glycol (EG), and ethanol (EtOH). The reduced viscosity of the copolymer is highly dependent on the ionic strength of the copolymer solution. The reduced viscosity decreases as a function of solvent selection in the order $W > DMSO > EtOH > EG$. The shapes of the η_{sp} / C vs. C plots indicate the polyelectrolyte behavior of the copolymer, except for the case of EG solutions, where nonpolyelectrolyte behavior is observed. However, at a certain degree of ionization attained by adding W as cosolvent, the copolymer begins to demonstrate polyelectrolyte behavior. For this copolymer, there exists a minimum concentration of brine (NaCl, CaCl₂, etc.) above which solution viscosity is not further reduced. The copolymer solution behaves as a power law fluid, and exhibits time-dependent thixotropic behavior. The copolymer cannot regain its solution viscosity when allowed to shear at a constant rate for long period of time. The reduced viscosities of copolymer solutions increase with increasing temperature in W and DMSO, yet decreases with increasing temperature in EG. © 2002 Wiley Periodicals, Inc. *J Appl Polym Sci* 85: 1521–1529, 2002

Key words: polyelectrolyte; water soluble polymers; rheology; shear

INTRODUCTION

In recent years there has been a considerable interest in applying water-soluble polymers in a variety of end uses.^{1,2} In addition to their use as flocculants for numerous substrates and as adhesives, they also constitute efficient thickeners/mo-

bility control agent in oil recovery techniques.^{3–5} High molecular weight ($>10^6$) partially hydrolyzed polyacrylamide (PHPA) is most commonly used as mobility control agent in polymer augmented or surfactant–polymer flooding techniques. PHPA suffers excessive thermal hydrolysis at high temperature, and as a result, may precipitate in presence of bivalent cations present in the reservoirs.⁶ So it cannot be applied to deep reservoirs where high temperatures exist. Copolymers of acrylamide with sulphonated comono-

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Table I Synthesis and Characteristics of the Copolymer

Parameters		Experimental Values	
Synthesis	wt % of AMPS in reaction mixture	30	
	Initiator conc. (%w/w of monomer)	(NH ₄) ₂ S ₂ O ₈	0.23
		Na ₂ S ₂ O ₅	0.33
	Reaction temperature (°C)	15	
	Reaction pH	1	
Reaction time (h)	4		
Characteristics	Intrinsic viscosity in 0.1 M aqueous NaCl solution at 25°C (dL/g)	4.58	
	wt % of AMPS in copolymer	26.62	
	Average molecular wt	M_n	0.52×10^6
		M_w	1.89×10^6
	Polydispersity	3.607	

mers, especially sodium 2-acrylamido-2-methylpropanesulphonate offer hydrogen bonding capability and polyelectrolyte behaviour in aqueous solution—characteristics of special interest in enhanced oil recovery (EOR).^{7,8} The polymer solution must be stable in harsh conditions of salinity and temperature prevailed in the reservoir. It is reported that NaAMPS does not precipitate in presence of CaCl₂.⁹ Another important criteria is the rate of shear encountered during injection of polymer solution. Efficiency of any water-soluble polymer for EOR depends on the effect of temperature, shear degradation, and viscosity loss in brine. For successful recovery the viscosity of the polymer solution must be equal to or greater than that of the oil to be displaced. In the present work, copolymer of *N,N*-dimethylacrylamide and sodium 2-acrylamido-2-methylpropanesulphonate is prepared, and the rheological behavior in four different solvents (water, ethylene glycol, dimethyl sulfoxide, and ethyl alcohol) is studied. The effects of shear, brine, and temperature on the solution viscosity of the copolymer are evaluated.

EXPERIMENTAL

Materials

Copolymer of *N,N*-dimethylacrylamide and sodium 2-acrylamido-2-methylpropanesulphonate is prepared in the laboratory. *N,N*-dimethylacrylamide (NNDAM, Aldrich) is exposed over calcium hydride for about 24 h to remove the stabilizers and then purified by distillation under vacuum. 2-Acrylamido-2-methylpropanesulphonic acid (AMPS,

Merck-Schuchardt) is purified by recrystallizing from methanol. Ammonium persulphate and sodium metabisulphite (CDH, New Delhi) are purified by recrystallizing from 60% ethanol–water mixture. Commercially available DMSO, EG, and EtOH are used after distillation. Water is distilled twice, boiled and cooled to room temperature before use. To get the degassed water nitrogen is passed for one hour. Specific conductance of the water used is 1.2×10^{-6} S cm⁻¹.

Preparation of Polymer

Synthesis of *N,N*-dimethylacrylamide and sodium 2-acrylamido-2-methylpropanesulphonate copolymer has been reported previously.^{9–11} The parameters of synthesis are given in Table I. Distilled NNDAM and purified AMPS monomers are dissolved in degassed, distilled water to get a 14% solution and taken in a three-necked flask fitted with a mechanical stirrer, a gas inlet tube, and a thermometer pocket. Residual oxygen is removed by bubbling nitrogen through the solution for an hour under constant stirring at 15°C. The reaction flask is allowed to stand in a large thermostatic bath maintained at a constant temperature of 15°C. Recrystallized ammonium persulphate and sodium metabisulphite solutions are added slowly, and the reaction is then allowed to proceed at that temperature for 4 h. The final solution is clear and highly viscous. The solution is diluted to three times its original volume. An aliquot of the solution is taken, and the polymer formed is isolated by precipitating into acetone and drying in a vacuum oven at 50°C for 15 h. The AMPS content in the copolymer is then determined by potentiometric titration.⁹ The remaining part of the reac-

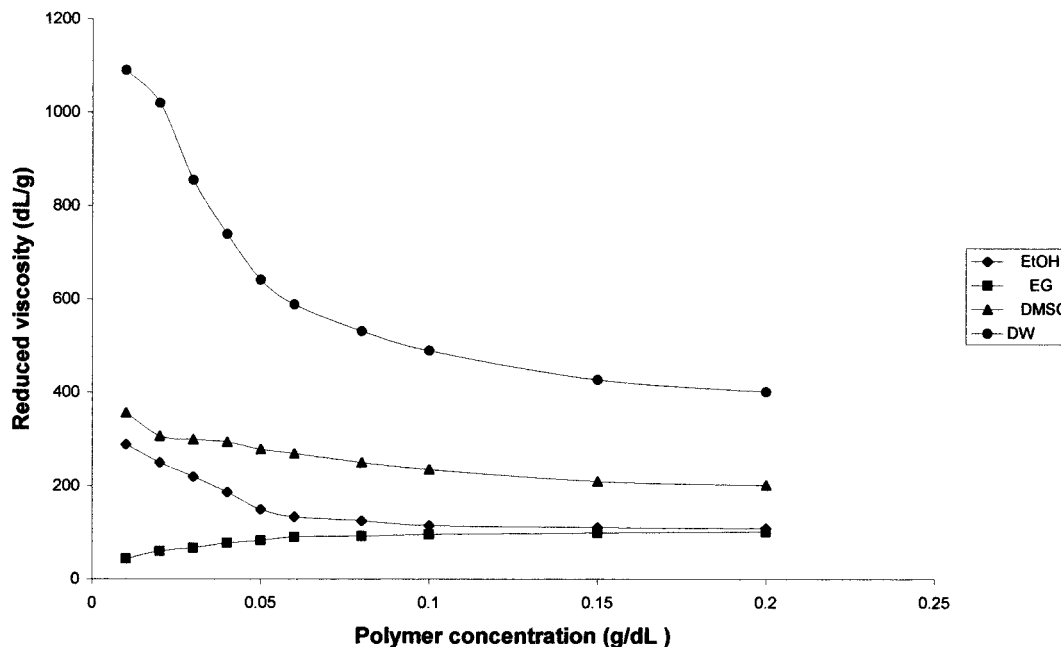


Figure 1 Concentration dependence of reduced viscosity in W, EG, EtOH, and DMSO at 7.34 s^{-1} and 25°C .

tion mixture is then neutralized with NaOH (2% solution). The final copolymer is isolated by precipitating into acetone and drying in a vacuum oven at 50°C for 15 h.

Polymer Solution Preparation

Appropriate quantities of polymer and solvent are taken in a closed vessel and kept overnight for dissolution. It is then stirred with a magnetic stirrer with minimum speed for 10–15 minutes, by the time clear solution is obtained. The solution is then filtered through a 100 mesh stainless still sieve.

Measurements

Intrinsic viscosity is evaluated by measuring the flow time of the polymer solution and the solvent using an Ubbelohde viscometer fitted in a Schott Gerate AVS 400 automatic instrument. Solution viscosities at low shear rate (up to 73.44 s^{-1}) are measured with a Brookfield LVTDV II viscometer equipped with UL adapter. Solution viscosities at high shear rate (up to 1312 s^{-1}) are measured with a Rotoviscometer. pH and potentiometric measurements are done at 25°C in a digital pH and mV instrument (Systronics Digital pH meter 335) assembled with a glass electrode and a calomel electrode. Conductivity measurements are

done by a Toshniwal digital conductivity meter and a conductance cell having a pair of platinum electrodes (cell constant 0.908 cm^{-1}). Molecular weight is determined at 25°C by Water's gel permeation chromatography equipped with a refractive index detector using Shodex OH Pak columns KB 802.5, KB 803, KB 804, and KB 806 and Polyacrylamide as calibrants.

RESULTS AND DISCUSSION

Table I presents the synthesis and characteristics of the copolymer. The number average molecular weight of the copolymer is 0.5×10^6 and polydispersity is 3.6. AMPS content of the copolymer is 26.62 wt %. Intrinsic viscosity is evaluated by extrapolation method using aqueous copolymer solution in 0.1 M NaCl at 25°C . The concentration dependence of reduced viscosity in four different solvents, (a) W, (b) DMSO, (c) EG, and (d) EtOH are presented in Figure 1. According to the accepted view, the reduced viscosity of polyelectrolyte solution decreases with increasing content of organic solvent. In line with this view, the reduced viscosity of the polymer solution in water is the highest. Fuoss et al.¹² observed a decrease in viscosity of a methanol–water solution of poly(vinyl butylpyridinium) bromide with an increase in

Table II Dielectric constants^a of Solvents and Specific Conductance of 0.02% Polymer Solution in the Solvents (at 25°C)

Solvent	Dielectric Constants of Solvents	Specific Conductance of Polymer Solutions (10^{-6} Scm^{-1})
W	78.5	36.5
EtOH	25.7	5.28
DMSO	48.9	6.79
EG	41.2	3.86
60%EG+40%W	57.7	9.32
40%EG+60%W	65.6	17.4
20%EG+80%W	72.2	28.6

^a Dielectric constants for EG-W mixtures are obtained from ref. 15.

the methanol concentration. A similar behaviour was also observed by Basu and Gupta¹³ for a dioxane–water solution of sodium carboxymethyl cellulose. The viscosity decrease was accounted for in terms of the decrease of the dielectric constant of the solvent, ϵ , with an increase in methanol concentration. As ϵ decreases, counter ions are associated closely with the polyion and the intramolecular electrostatic repulsive interactions between the ionized groups is diminished allowing the chain to form a more compact coil. Yamanaka et al.¹⁴ also observed the decrease of reduced viscosity of polystyrene sulphonates with increasing EG content in the aqueous solvent. This was accounted for in terms of hydrodynamic volume.

If the dielectric constant is the main criteria vis-à-vis polyion shrinkage, the reduced viscosity of the polymer solution is expected to be greater in EG than in EtOH, where the dielectric constant is the lowest. However, this is not observed, the reduced viscosity in EtOH is greater than that in EG solution. For polyelectrolytes, solution viscosity is greatly influenced by the overall ionic strength and polyion concentration¹⁵ of the solution (i.e., polyion–counterion and polyion–polyion electrostatic interactions). Dielectric constants of solvents and specific conductance of the polymer solution in different solvents are presented in Table II. The specific conductance of the polymer solution decreases in the order $W > \text{DMSO} > \text{EtOH} > \text{EG}$, as does the trend for the reduced viscosities of the polymer solutions.

Ethylene glycol is a highly polar solvent, having strong hydrogen bonds with two OH groups. It is observed that EG takes longer time to dissolve

a certain amount of polymer than EtOH. As it is a poorer solvent, the interactions between the polymer and the solvent molecules is weaker in comparison to that in EtOH. Probably this is due to the stronger H-bonding between the EG molecules. So, the polyion–counterions are closely associated, diminishing the third-order electroviscous effect in the polymer molecule, i.e., diminishing the intramolecular electrostatic repulsive interaction between the ionized groups resulting the decrease of hydrodynamic volume and, hence, the reduced viscosity. It may be mentioned that the molar volume of EtOH (58.5 mL/mol) is also greater than that of EG (55.8 mL/mol).

The increase in reduced viscosity with dilution in W, DMSO, and EtOH is due to the increase of degree of dissociation of the polyelectrolyte with dilution. As discussed above, polyion–counterions are closely associated in EG reducing the hydrodynamic volume. Upon dilution, a decrease of the polymer intermolecular interaction and hence further decrease of hydrodynamic volume occurs. This results in the decrease of reduced viscosity, and the polymer solution exhibits nonpolyelectrolyte behavior. It is observed from Table I that both dielectric constant as well as the specific conductance increases upon the addition of water to EG. The intramolecular electrostatic repulsive interaction between the ionized groups will slowly become significant upon addition of water. This results in the increase of hydrodynamic volume, and hence the reduced viscosity exhibits polyelectrolyte behavior. There appears to be a limiting concentration of EG beyond which the shape of the η_{sp}/C vs. C curve changes from one type to another (Fig. 2). In EG-W(60–40%) solvent the curve represents nonpolyelectrolyte behavior. But upon increasing water content, i.e., in EG-W(40–60%) solvent, the polyelectrolyte behavior appears. In the DMSO-W and EtOH-W system the η_{sp}/C vs. C curves follow the polyelectrolyte behavior (Figs. 3 and 4).

Effect of Saline Solvent

In dilute, salt-free water solution the polymer molecules expand, largely due to the smaller volume interaction between the charges on the backbone of the polymer. At low ionic strength and high charge density, the electrostatic repulsive forces dominate the polymer solution behavior.¹⁶ If an electrolyte is added to the solution, the negative charges on the polymer moiety are shielded, resulting in the reduction of repulsive interac-

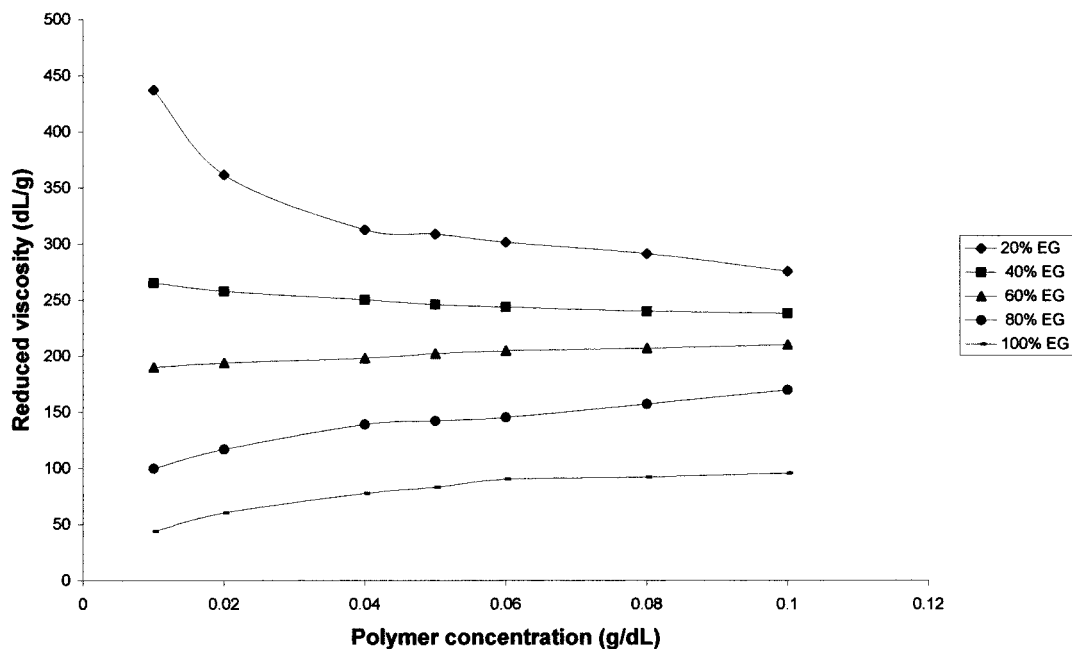


Figure 2 Concentration dependence of reduced viscosity in EG–W mixed solvent at 7.34 s^{-1} and 25°C .

tion.¹⁷ The molecule tends to contract in solution, resulting in the decrease of solution viscosity. This effect is more pronounced for bivalent cat-

ions than that for monovalent cations. It can be observed from Figure 5(a) and (b) that there exists an optimum limit of the concentration of

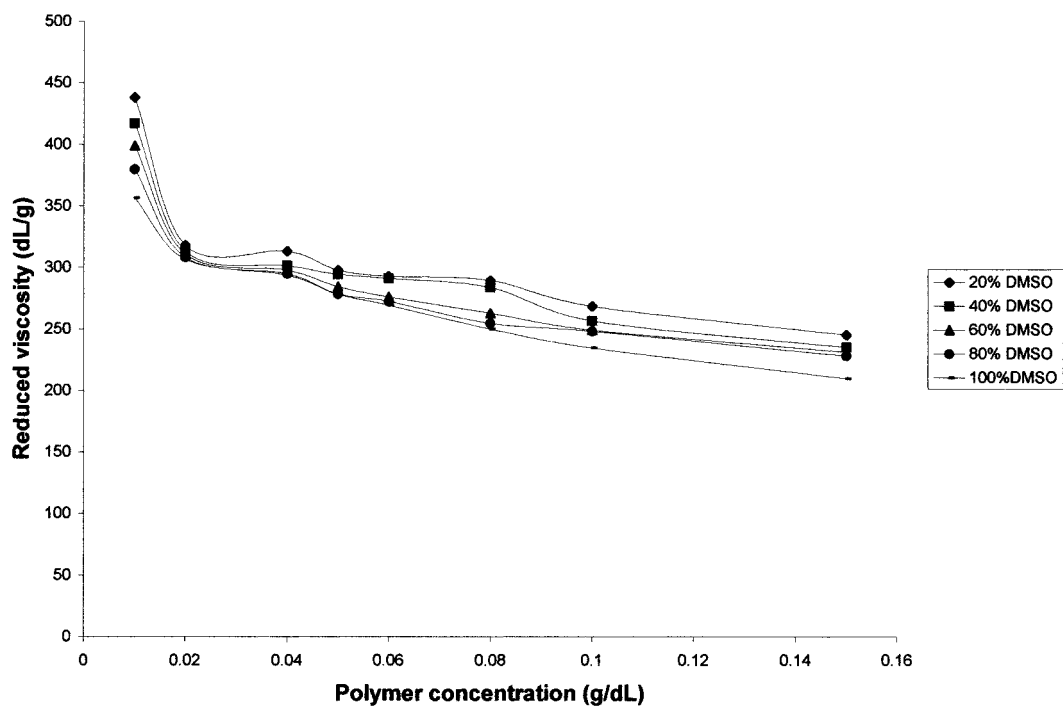


Figure 3 Concentration dependence of reduced viscosity in DMSO–W mixed solvent at 7.34 s^{-1} and 25°C .

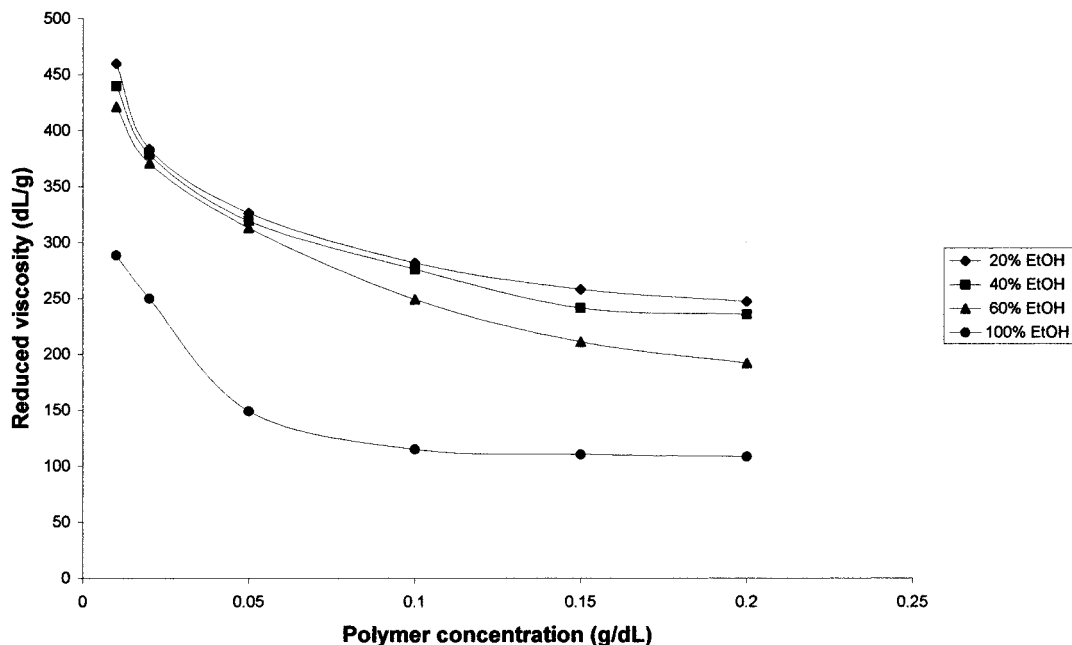


Figure 4 Concentration dependence of reduced viscosity in EtOH-W mixed solvent at 7.34 s^{-1} and 25°C .

NaCl or CaCl_2 , beyond which it can not reduce the solution viscosity any more. It also reveals that after optimum reduction the solution viscosity is the same irrespective of monovalent or bivalent brines. It is reported^{14,18} that polystyrene sulpho-nate and partially hydrolyzed polyacrylamide exhibit Newtonian behavior at high concentration of NaCl in EG solution. With the addition of NaCl, the decrease of solution viscosity is observed, but this copolymer exhibits non-Newtonian behavior.

Effect of Shear

The rheological plot of $\log \eta$ vs. $\log \dot{\gamma}$ is presented in Figure 6. It is observed that a linear correlation of $\log \eta$ and $\log \dot{\gamma}$ exists at least up to a shear rate of 1312 s^{-1} (studied), showing the system to obey the “power law,” i.e., $\eta = K(\dot{\gamma})^n$, where K is the consistency and n is the power law index that measures the pseudoplasticity of the polymer solution. If $n < 1$, the apparent viscosity of a pseudoplastic fluid decreases with increasing shear rates, i.e., it is rheopectic fluid. If $n = 1$, the polymer solution obeys the Newtonian flow behavior.

The calculated K and n values for the copolymer at a narrow range of shear rate ($0\text{--}73.44 \text{ s}^{-1}$) are presented in Table III. It shows that the value of K increases with the increase of EG content of

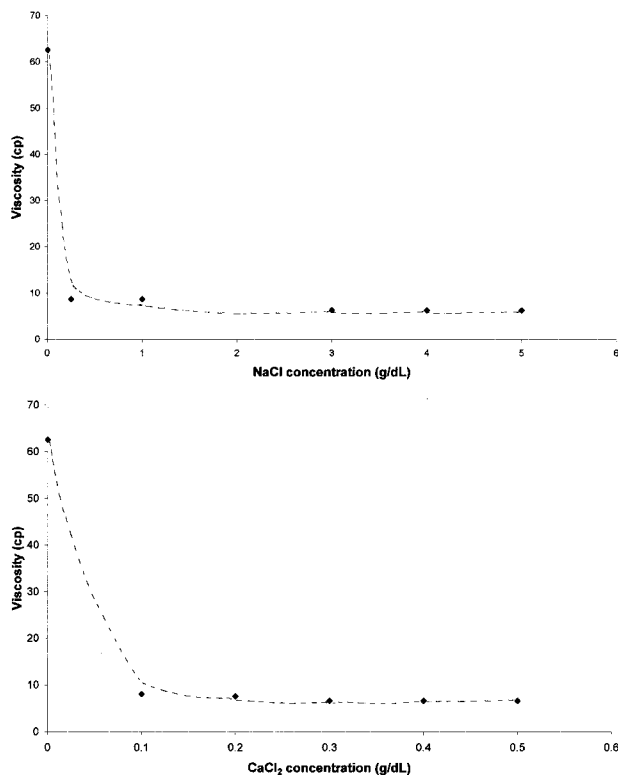


Figure 5 (a) Effect of NaCl concentration on solution viscosity of 0.2% aqueous polymer solution at 7.34 s^{-1} and 25°C . (b) Effect of CaCl_2 concentration on solution viscosity of 0.2% aqueous polymer solution at 7.34 s^{-1} and 25°C .

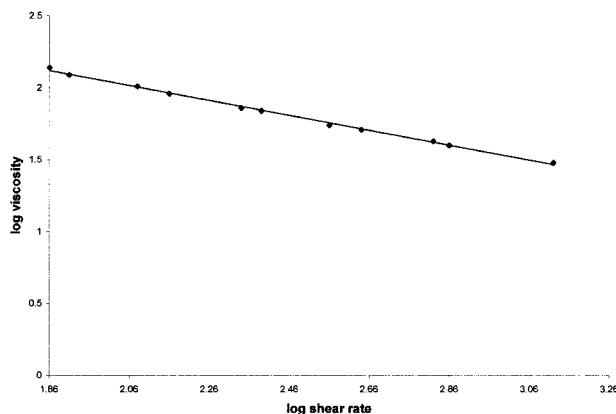


Figure 6 Rheological plot of log viscosity vs. log shear rate for 0.8% aqueous polymer solution (shear rate range $72.9\text{--}1312\text{ s}^{-1}$) at 25°C .

the solvent. The addition of EG also increases the value of n , i.e., approaching unity. So, the addition of more and more quantity of EG, the polymer molecular behavior approaches to the Newtonian value. But for DMSO and EtOH, the value of n is more or less constant, irrespective of their concentration. The value of n , i.e., pseudoplasticity of the polymer solution is found to be same for W and EtOH followed by DMSO and EG for 200 ppm of the polymer. The effect of temperature on pseudoplasticity is found to be nil.

It is also observed that in presence of NaCl, the polymer solution obeys the power law in a limited range of shear rate. At low shear rates ($0\text{--}15\text{ s}^{-1}$) the value of n is more deviated from unity showing perfectly non-Newtonian nature. But in higher range of shear rates ($36\text{--}73.44\text{ s}^{-1}$) the value of n approaches unity in both water and EG solution. This indicates that at higher shear rates, the rheological behavior of the polymer solution approaches Newtonian characteristics.

When the polymer solution is continuously sheared at a constant rate, the structure progressively disintegrates and the apparent viscosity decreases with time. The rate of structure breakdown during shearing at a given rate depends on the number of linkages available for breaking and must, therefore, decrease with time. The simultaneous rate of reformation of structure of the polymer will increase with time as the possibility of new structural linkages increases. Eventually, a state of dynamic equilibrium is achieved when the rate of build up of structure equals the rate of breakdown.¹⁹ This equilibrium position depends on the rate of shear and moves toward greater breakdown at increasing shear rate. The polymer solution in W, EG, and DMSO are sheared continuously at a constant rate to achieve the equilibrium viscosity (Fig. 7). It is then allowed to stand without shear for 10 h. The polymer solution try to regain the original viscosity slowly. As presented in the Figure 7, viscosity regain is partial irrespective of the solvent. This shows that the polymer solution undergo deformation when subjected to shear, however, part of their deformation is gradually recovered, if not fully. It is also observed that the solution viscosity remains constant for the same period if it is allowed to stand without shear. The irrecoverable loss may be due to the mechanical degradation of the polymer under shear. Similar behavior is observed for viscoelastic fluids, which cannot recover its original viscosity after deformation.²⁰ In a separate experiment it is observed that the same polymer solution in presence of brine (10,000 ppm NaCl) can regain its initial viscosity after keeping only 7 h without shear after shearing for 10 h. In presence of NaCl, polymer molecule tends to contract in solution resulting the decrease in solution

Table III Power Law Constants for 0.02% Polymer in Different Solvents at a Shear Rate Range of $0\text{--}73.44\text{ s}^{-1}$, 25°C

Solvent	K (mPas ⁿ)	n	Solvent	K (mPas ⁿ)	n
W	43.65	0.57	40%EG+60%W	29.85	0.67
EtOH	28.84	0.5	20%EG+80%W	29.85	0.64
60%EtOH+40%W	43.15	0.69	DMSO	52.48	0.69
40%EtOH+60%W	42.66	0.67	80%DMSO+20%W	51.29	0.67
20%EtOH+80%W	34.67	0.64	60%DMSO+40%W	22.91	0.87
EG	65.01	0.79	40%DMSO+60%W	33.88	0.64
80%EG+20%W	41.69	0.79	20%DMSO+80%W	22.38	0.67
60%EG+40%W	41.69	0.67			

viscosity. Due to the reduction of the hydrodynamic volume of the polymer, further degradation under shear will be minimum.

Effect of Temperature

The plots of reduced viscosity vs. temperature of the polymer solution in W, DMSO, and EG solvents are presented in Figure 8. The reduced viscosity increases with the increase of temperature in W and DMSO solvents. As temperature increases, the macroions will expand due to the increased degree of dissociation of the ionizable groups producing larger hydrodynamic volume resulting the increase of reduced viscosity. In EG, the polyion-counterions are closely associated, reducing the intramacroion repulsive electrostatic interactions. The reduced viscosity of the polymer in EG is, therefore, mostly due to the intermolecular association between the polymer molecules. As temperature increases, both the polymer chain stretching and decrease of intermolecular association will take place. The first one increases the reduced viscosity, while the second one reduces it. It is observed from Figure 8 that no change in reduced viscosity occurs up to a temperature of 60°C, above which a slight decrease appears. This may be due to the predominate effect of the second phenomena over the first one. It may be mentioned that every care is taken to keep the volume of the experimental liquid constant.

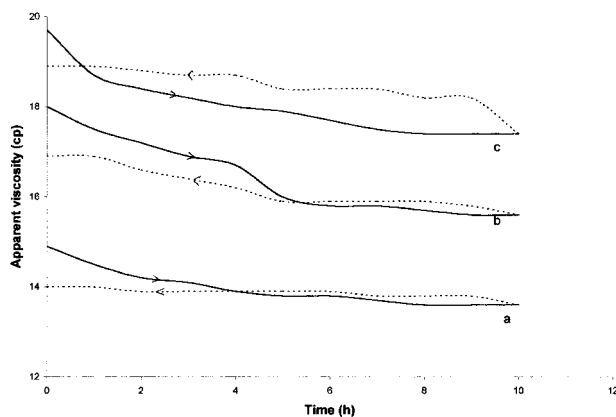


Figure 7 Time dependent hysteresis loop for (a) 0.05% polymer in W at 35°C, (b) 0.02% polymer in EG with 0.02% NaCl at 30°C, (c) 0.02% polymer in DMSO at 30°C under dynamic (bold line—constant shear rate of 36.72 s^{-1}) and static condition (dashed line).

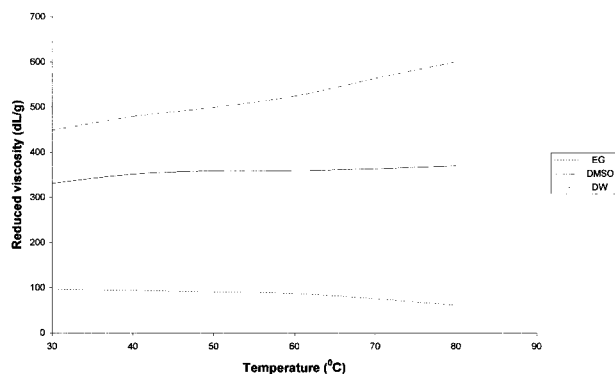


Figure 8 Effect of temperature on reduced viscosity of 0.05% polymer solution in W, DMSO, and EG at 7.34 s^{-1} .

CONCLUSION

Copolymer of *N,N*-dimethylacrylamide and sodium 2-acrylamido-2-methylpropanesulphonate exhibits polyelectrolyte behavior in W, DMSO, and EtOH solvents, but in EG solvent it exhibits nonpolyelectrolyte behavior. The reduced viscosity of the polymer solution follows the order of specific conductance of the different solutions. The shape of the plots of η_{sp}/C vs. C is quite opposite for EG solution to that for the other solutions. In EG–W solution, there appears to exist a certain degree of ionization, below which the polymer solution shows nonpolyelectrolyte behavior. Solution viscosity decreases with the increase of shear rate, i.e., pseudoplastic behavior. It obeys power law as well as time-dependent thixotropic behavior. It cannot regain its solution viscosity when it is allowed to shear for a long period of time. In the presence of brine, it obeys power law in a limited range of shear rate, but it can regain its solution viscosity fully, that lost during shearing for a period. With the increase of temperature, reduced viscosity of the dilute polymer solution increases in W and DMSO, but decreases in EG solvent.

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