Effect of Inorganic Salts on Viscosifying Behavior of a Thermoassociative Water-Soluble Terpolymer Based on 2-Acrylamido-methylpropane Sulfonic Acid

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ABSTRACT: The effect of different inorganic salts on aqueous solution viscosifying behavior of a thermoassociative water-soluble terpolymer, poly(acrylamide-co-2-acrylamido-2-methylpropane sulfonic acid)-g-poly[acrylamide-co-N-(1,1-dimethyl-3-oxobutyl)acrylamide] (PADAS) was examined in comparison with a nonthermoassociative sulfonated copolymer, poly(acrylamide-co-2-acrylamido-2-methylpropane sulfonic acid) (PAMS). With increasing salt concentration, thermoviscosifying ability of PADAS was clearly evidenced and the magnitude of thermoviscosifying effect was largely enhanced. The ability of cations and anions to enhance the viscosity of PADAS aqueous solution follows the orders $Na^+ > K^+ > Mg^{2+} > Ca^{2+}$ and $SO_4^{2-} > Cl^- > Br^- > NO_3^-$, respectively. Under the same conditions, PAMS shows only thermothinning behavior and viscosity loss. The thermoviscosifying capability of PADAS is relevant to the change in the polarity of aqueous solution and the electrostatic shield effect of counterion for the repulsion between the sulfonate anions on the polymer, and depends mainly on the competition of these two effects. Cryo-TEM observation demonstrates that the microstructure changes of polymers in the electrolyte solution lead to the changes of the viscosifying behavior. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

Key words: thermoassociative; viscosifying behavior; water-soluble polymer; polyelectrolyte; viscosity; inorganic salt

INTRODUCTION

Partially hydrolyzed polyacrylamide (HPAM) is one of the most widely used and technically important water-soluble polymeric materials[1] and has found various applications ranging from flocculation,[2] mineral processing,[3] soil conditioning,[4] dispersion,[5] shale stabilization,[6] drilling fluid additives,[7] to tertiary oil recovery.[8–10] When used in petroleum industry, nevertheless, large reduction in HPAM solution viscosity is evidenced because of the presence of electrolytes and increased temperature in the oil reservoirs,[11,12] as the amide groups undergo extensive

hydrolysis into carboxylic moieties at elevated temperature, and the resulting hydrolyzed products precipitated when contacting divalent ions, Ca^{2+} and Mg^{2+}, commonly present in oil reservoir brines or hard water.[11–14] Thus, efficient water-soluble polymers for producing oil from high-temperature, high-salinity oil reservoirs are urgently demanded, and great efforts from various laboratories have been made to improve HPAM polymers by copolymerization of acrylamide with more thermally stable and salt-tolerant monomers to minimize excessive hydrolysis and thus enhanced thermal stability of the products.[15–20]

As a well-known salt-tolerant and thermostable comonomer, 2-acrylamido-methylpropane sulfonic acid (AMPS), has received much attention in the past decades,[21,22] and has been frequently incorporated onto the skeleton of water-soluble copolymers such as poly[AMPS-co-AA(acrylic acid)],[21] poly [AMPS-co-MA(maleic acid)],[22] and poly(AMPS-co-AM),[23] to name just a few. It is found that the copolymer of AM and sodium salt of AMPS (NaAMPS), PAMS, with a specific sulfonation degree showed better calcium tolerance than HPAM,[23] and the
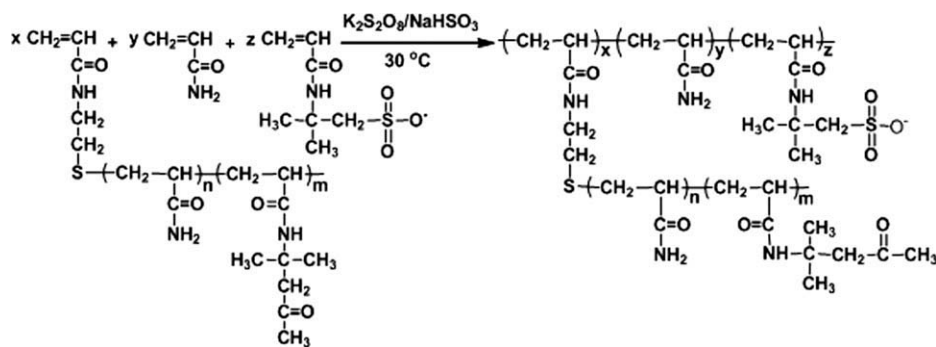
Figure 1 Synthesis route of thermoassociative polymer PADAS.

hydrolytic stability of the copolymer containing 40 mol % AMPS is superior to that of HPAM when aging at 93°C for more than 100 days.[24] Nevertheless, the apparent viscosity of PAMS aqueous solution still tends to decrease at high salinity and elevated temperatures, and the AMPS group may also hydrolyze at temperature higher than 100°C.[25,26]

To obviate the above limitations of HPAM and PAMS polymers, Hourdet et al.[27,28] developed "thermoassociative" water-soluble polymers mainly based on thermosensitive poly(N-isopropylacrylamide) (PNIPAM) and polyethylene (PEO). A well-studied example of such associative copolymer is poly (AMPS-co-AA)-g-PEO[28] that showed good thermal and hydrolytic stability, as well as good tolerance to KCl.[28] However, expensive coupling agent is obligatorily necessary in the preparation of polymers, and the grafting reaction is always conducted at relative low polymer concentrations.[27–30] Most importantly, some specific inorganic salt, particularly K_2CO_3 is always needed to induce the thermoassociation between the grafts.[29] Considering the target applications in oil industry, to the best of our knowledge, there is no report to date examining the influences of different ionic species particularly divalent cations in the oil reservoirs on the viscosity behaviors of these thermoassociative polymers.

An attempt has been made in our laboratory to prepare novel thermoassociative polymers by copolymerization of acrylamide, AMPS, and our newly-developed thermosensitive comonomer based on N-(1,1-dimethyl-3-oxobutyl)-acrylamide (DAAM) with more convenient and cost-effective procedures.[31,32] Presented in this work are the comparative results of the effect of different inorganic salts on the viscosity properties of the thermoassociative terpolymer and nonassociative copolymer.

EXPERIMENTAL

Materials

Acrylamide (AM, 99.5%, Changjiu Agri-Scientific Co. Ltd, Nanchang, China) and 2-acrylamido-2 methyl-propane sulfonic acid (AMPS, 99.2%, Shandong Lianmeng Chemical Co. Ltd, China) were used as received. Potassium persulfate (KPS), sodium bisulfite, and other solvents were all analytical grade, and obtained from Shanghai Chemical Reagent Co. Ltd, China. The water used was doubly distilled with an all-glass apparatus, and N_2 has the purity of 99.999%. The synthesis and characterization of macromonomer MPAD based on N-(1,1-dimethyl-3-oxobutyl)-acrylamide) were reported previously.[31,32]

Preparation of polymers

The terpolymer, poly(acrylamide-co-2-acrylamido-2-methylpropane sulfonic acid)-g-poly[acrylamide-co-N-(1,1-dimethyl-3-oxobutyl)acrylamide], PADAS, was prepared by direct free radical copolymerization of AM, AMPS, and macromonomer MPAD in pure water (Fig. 1): MPAD (2.50 g), AM (5.50 g), and AMPS (2.00 g) were dissolved in 90 mL water, and the pH was adjusted around 8 using NaOH followed by 30 min of N_2 purge. Then 0.5 mL of 1% (w/v) KPS and 1% (w/v) sodium bisulfite aqueous solutions were injected, respectively. The polymerization was carried out at 30°C under N_2 atmosphere for 10 h. The resulting product was obtained by dropwise addition into fivefold excess of acetone, and repeatedly washed with acetone, followed with freeze-drying, and 7.60 g of the final terpolymer product was obtained.

For comparison, the copolymer, poly(acrylamide-co-2-acrylamido-2-methylpropane sulfonic acid), PAMS, was prepared under the identical conditions. The intrinsic viscosity ([η]) of both PADAS and PAMS determined in $1M$ NaCl aqueous solution by an Ubbelhode capillary glass viscometer (ID = 0.56 mm) was 351 mL g^{-1} and 1150 mL g^{-1}, respectively.

Characterization of polymers

^1H NMR spectra of both PADAS and PAMS were recorded on a 300 MHz Bruker spectrometer in D_2O. As shown in Figures 2 and 3, the NMR signal
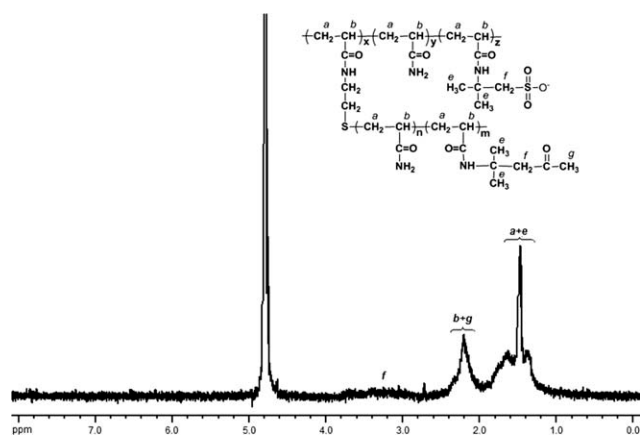
Figure 2 ^{1}H NMR spectrum of thermoviscosifying copolymer PADAS.

TABLE I
Physical Parameters and Composition of Copolymers

Polymer	Feed ratio (wt %)			Final composition (wt %)[a]			$[\eta]$ (mL g^{-1})
	AM	AMPS	MPAD	AM	AMPS	MPAD	
PAMS	75.0	25.0	0	69.2	30.8	0	1.15×10^{3}
PADAS	55.0	25.0	20.0	54.2	27.5	18.3	3.51×10^{2}

[a] Elemental analysis results

together with their allocation were attributed to different protons, particularly the characteristic peaks at 3.21 ppm ($-CH_2CO-$ and $-CH_2SO_3^{-}-$), 2.1 ~ 2.41 ppm ($-COCH_3$ and $-CHCO-$), 1.36 ~ 1.81 ppm ($-CH_2$ and $-CH_3$). However, it was difficult to quantitatively determine the final compositions of copolymers with ^{1}H NMR because of the complex molecular structures and the ambiguous integrals in the spectra, and thus elemental analysis was employed to help more accurate determination of the polymer compositions following a previously reported procedure.[31] Both feed ratio and calculated final mass percentages of AM, AMPS, and MPAD in the polymers were listed in Table I.

Apparent viscosity measurement

Different concentration of polymer solutions were prepared by dissolution of an appropriate amount of polymer powders in distilled water or different brines with gentle magnetic stirring. The solutions were then allowed to stand for 1 day until any bubbles present disappeared. Apparent viscosities of
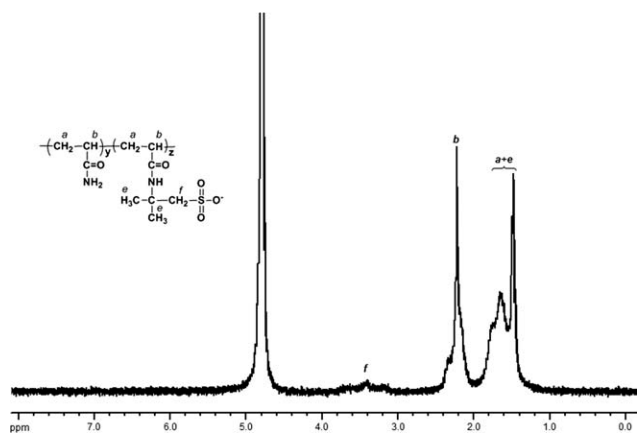
polymer solutions were measured at a constant shear rate of 10 s^{-1} during temperature scans going from 25°C to 95°C with a Brookfield LVDV-III programmable viscometer equipped with SC4-31 spindle and corresponding adaptor. Temperature was regulated with a thermostatting water circulating bath and detected instantaneously using a temperature probe connected between adaptor and viscometer.

Cryogenic transmission electron microscopy observation

Cryogenic transmission electron microscopy (Cryo-TEM) observation of both PADAS and PAMS solutions was carried out in a controlled-environment vitrification system. The specimens were preheated to 80°C, and the relative humidity was kept close to saturation to prevent evaporation from the samples during preparation. Five microliters of the preheated sample solution was placed on a carbon-coated holey film supported by a copper grid and gently blotted with filter paper to obtain a thin liquid film on the grid. The grid was quenched rapidly in liquid ethane at −180°C and then transferred to liquid nitrogen (−196°C) for storage. The vitrified specimen stored in liquid nitrogen was transferred to a JEM2010 cryo-microscope using a Gatan 626 cryo-holder and its workstation. The acceleration voltage was 200 kV and the working temperature was kept below −170°C. The images were recorded digitally with a charge-coupled device camera (Gatan 832) under low-dose conditions with an under-focus of ~ 3 μm.

RESULTS AND DISCUSSION

Concentration dependence of the polymer solution viscosity

Compared in Figure are the viscosity variation of PADAS and PAMS in pure water with temperature for polymer concentrations (C_p) between 0.2% and 1.0% (w/w, the same below). As shown in Figure 4(a), when C_p of PADAS is lower than 0.2%, the apparent viscosity (η_{app}) decreases upon increasing

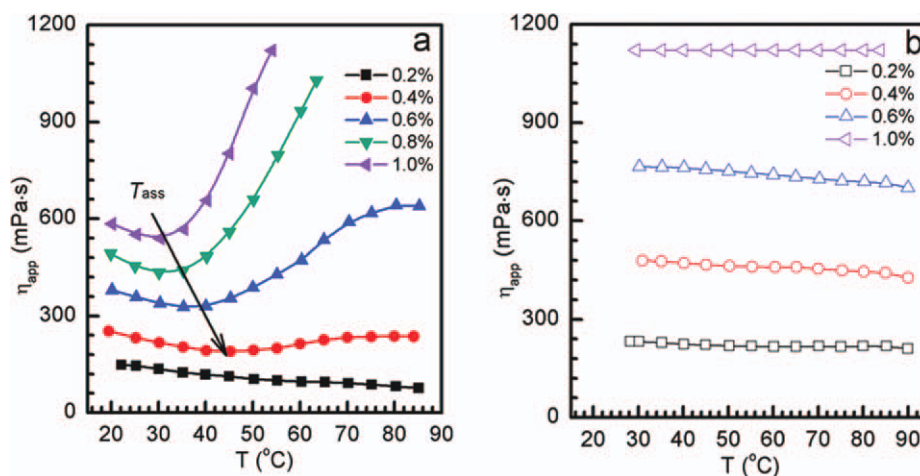**Figure 3** ^{1}H NMR spectrum of copolymer PAMS.

Figure 4 Apparent viscosity plotted as a function of temperature for (a) PADAS and (b) PAMS at different concentrations in pure water ($\dot{\gamma} = 10 \text{ s}^{-1}$). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

temperature, which represents a typical thermothinning behavior; however, when C_p is higher than 0.2%, η_{app} of PADAS solution increases with increasing temperature, and the thermoassociative temperature (T_{ass}) decreases when increasing polymer concentration, for example, T_{ass} is shifted from 48°C to 32°C when increasing C_p from 0.4% to 1.0%. Such a thermoviscosifying behavior of PADAS aqueous solutions is proportional to the thermosensitive sidechains, or the total number of physical crosslinks, thus the higher the polymer concentration, the stronger the thermoviscosifying ability is.[29,30] Nevertheless, the viscosity of PAMS aqueous solutions in all concentrations [Fig. 4(b)] decreases smoothly with increasing temperature, with a general trend following the Arrhenius law.

Effect of NaCl on polymer solution viscosity

Salts can change the structure of water and thus the free energy of interaction between the polymer and water. The change of water structure may strongly affect the thermoassociative behavior of thermoassociative water-soluble polymers. NaCl is a typical so-called water structure maker that promotes the hydration sheath near the polymer chains to become partially destroyed.[33] Figure 5 shows the influence of NaCl on the viscosifying behavior of PADAS and PAMS aqueous solutions within the temperature range from 25°C to 90°C. From the comparative results, the following distinctive features can be found: (i) η_{app} of PADAS in NaCl electrolyte solution remains nearly unchanged at low temperature, following a continuous increase when above T_{ass},
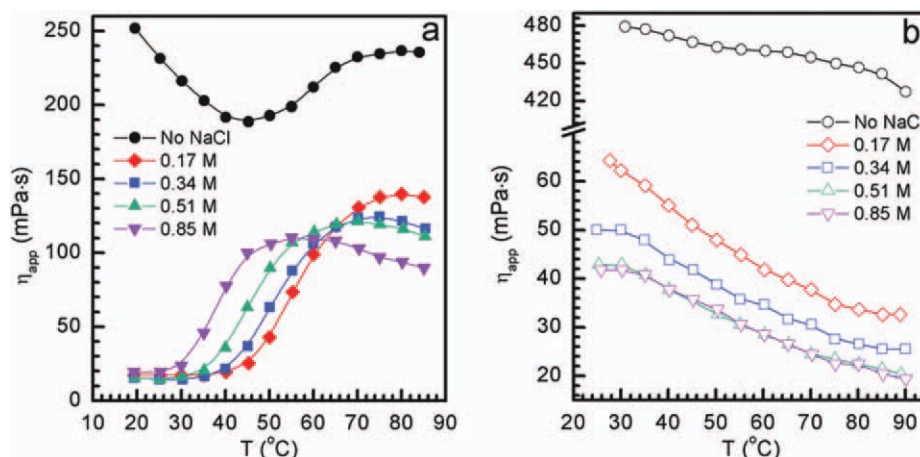

Figure 5 Apparent viscosity plotted as a function of temperature for (a) PADAS and (b) PAMS in different NaCl solutions ($C_p = 0.4\%$, $\dot{\gamma} = 10 \text{ s}^{-1}$). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
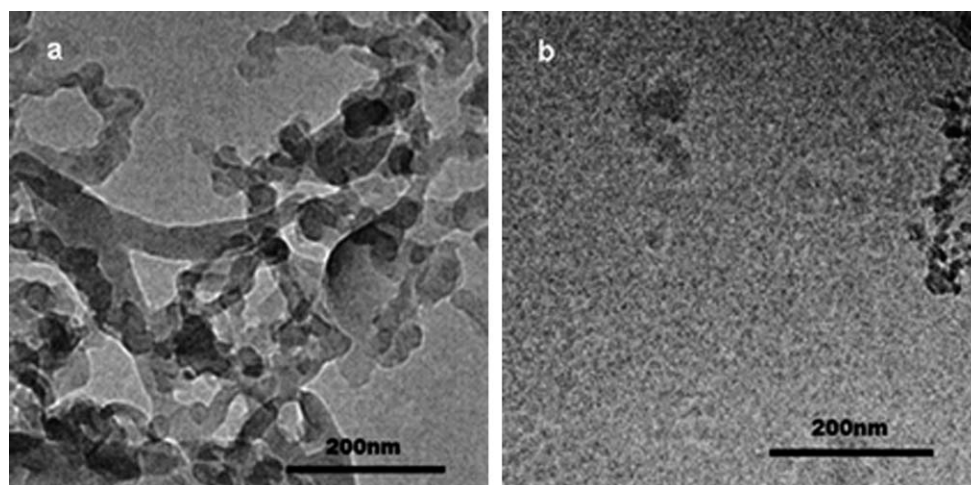
Figure 6 Cryo-TEM images of (a) PADAS and (b) PAMS in 5% NaCl solution ($C_p = 0.4\%$, $T = 80°C$). The scale bars are 200 nm.

however, η_{app} of PAMS solutions decreases upon increasing temperature; (ii) η_{app} of PADAS in NaCl electrolyte solution is lower than those in pure water, and the magnitude of thermoviscosifying effect was largely enhanced in such a electrolyte solution; (iii) T_{ass} of PADAS solutions decreases when increasing NaCl concentration, for example, for PADAS, when increasing NaCl concentration from 0 to $0.85M$, T_{ass} is lowered from 50°C to 44°C, and η_{app} in $0.85M$ NaCl electrolyte solution increases nearly 8.1 times between 25°C and 80°C, while η_{app} in pure water increases only 1.1 time in the same temperature range.

Ordinary anionic polyelectrolytes, such as HPAM and PAMS, exhibit larger solution viscosity than nonionic polymers as a result of repulsive interaction of groups with anionic charges along the polymer chain. The ionic repulsive forces increase hydrodynamic volume of anionic polyeletrolytes, particularly in pure water or low salinity electrolyte solution. An increase in salt concentration shields ionic charges which weakens the repulsive forces among the charges. At the same time, it must be pointed out that small and/or polyvalent ions are generally considered as "structure makers", and hydrogen bonding with the amide is partially destroyed, leading to the change in solubility and viscosity. Consequently, the hydrodynamic volumes of the polymer chains decrease, leading to the polymer coils collapse and viscosity loss macroscopically. But for thermoassociative polymer, PADAS, the addition of NaCl also enhances the intermolecular association because of the increased polarity of the solvent, since macromonomer side chains start to self-assemble into hydrophobic microdomains when above T_{ass}. When thermoassociation prevails over electrostatic screening, the apparent viscosity of PADAS aqueous solutions increases upon increasing

temperature. Thus, the thermoviscosifying response is the synergistic results of both charge shielding between sulfonated groups and thermoassociation between macromonomer side chains induced by the increased polarity.

To gain insight into the thermoassociative mechanism, direct visualization with Cryo-TEM was applied to both PADAS and PAMS aqueous solutions (Fig. 6). As shown in Figure 6(a), the self-assembling microdomains and the physical junctions of the network structures formed by PADAS solution are clearly visible; however, no such microstructures were observed in PAMS solutions [Fig. 6(b)]. When heated above the temperature higher than T_{ass}, water becomes a bad solvent for thermosensitive side groups and these side groups along PADAS skeleton start to gradually self-aggregate into hydrophobic microdomains, and the polymer mainchains can also entangle with each other, leading to the formation of a thermally induced three-dimensional network in PADAS semidilute aqueous solution (Fig. 7). Nevertheless, for the non-associative PAMS, only entanglements but no assembling structures were formed in this case, as there are no thermosensitive side chains in the polymer backbone. The microstructure changes of polymers in the electrolyte solution lead to changes in the viscosifying behavior.

Effect of anions on polymer solution viscosity

A given simple inorganic salt presumably exerts its effect on polymer solution viscosity through the combined effects of its anion and cation. To compare the relative importance of these contributions, the influence of a series of anions on polymer solution viscosity was firstly investigated. Figure 8 shows the variation of η_{app} with temperature for both PADAS
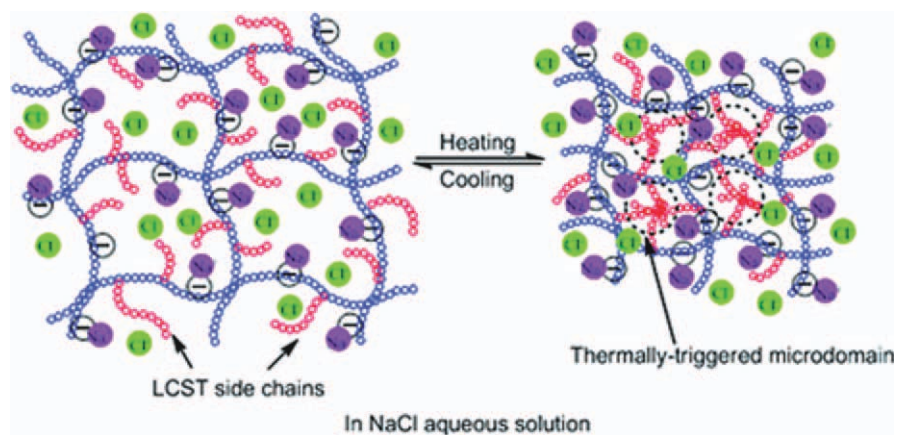
Figure 7 Schematic illustration of the effect of NaCl on the thermoresponsive aggregation for PADAS. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and PAMS in 0.2M different sodium salt aqueous solutions. As shown in Figure 8(a), PADAS aqueous solutions have a clear thermoviscosifying property and such a behavior strongly depends on anion species. The ability of anions to enhance the viscosity of PADAS aqueous solution nearly follows the order: $SO_4^{2-} > Cl^- > Br^- > NO_3^-$, which is in good agreement with the Hofmeister series.[34] However, no thermoviscosifying trend exists for PADAS solution in Na_2CO_3 electrolyte solution, which may be because of the hydrolysis of amide group on the macromonomers at high temperature leading to the loss of thermoviscosifying property. It is worth noting that salt effects on T_{ass} of PADAS almost correlate to the position of the corresponding salt in the classic Hofmeister series, which originally categorized salts in regard to their "salting out" potential toward proteins from aqueous solution.[34,35]

Figure 8(b) clearly indicates the slight anion salinity sensitivity of the apparent viscosity of PAMS. As

described earlier, when adding inorganic salt to PAMS solutions, the charges on the polymer chain are shielded and the molecules coil up. The electrostatic shield effect of inorganic salt depends strongly on the concentration and species of the counterion for the repulsion of ionic head group on the polymer chain, almost regardless of the other ion of inorganic salt.

Effect of cations on polymer solution viscosity

Exhibited in Figure 9 are the viscosity behaviors of PADAS and PAMS in 0.2M different chlorides electrolyte solutions upon heating. It was found that the thermoviscosifying ability of PADAS aqueous solutions also strongly depends on cation species. The typical order of the ability of cations to enhance the viscosity of PADAS solution is: $Na^+ > K^+ > Mg^{2+} > Ca^{2+}$. The is because the negative charges on the PADAS side chains could be effectively shielded by
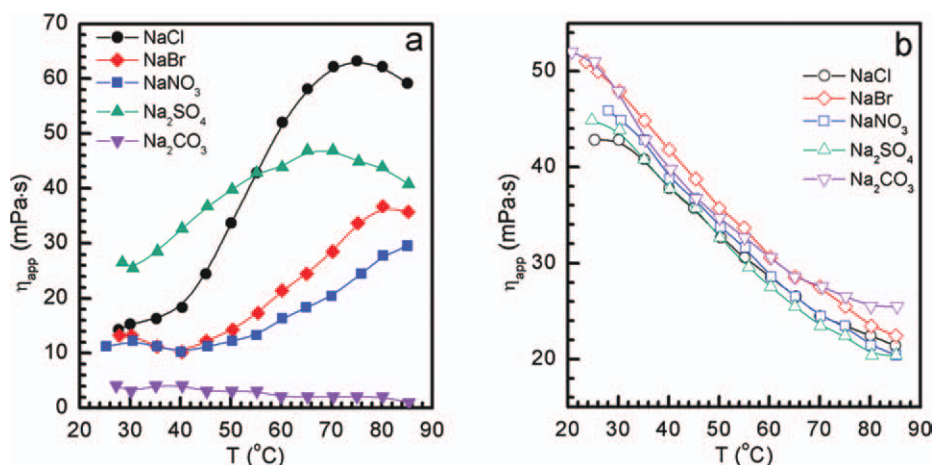

Figure 8 Apparent viscosity plotted as a function of temperature on polymer solution viscosity for (a) PADAS and (b) PAMS in different sodium salt brines ($C_p = 0.4\%$, anion concentration = 0.2M, $\dot{\gamma} = 10 \text{ s}^{-1}$). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
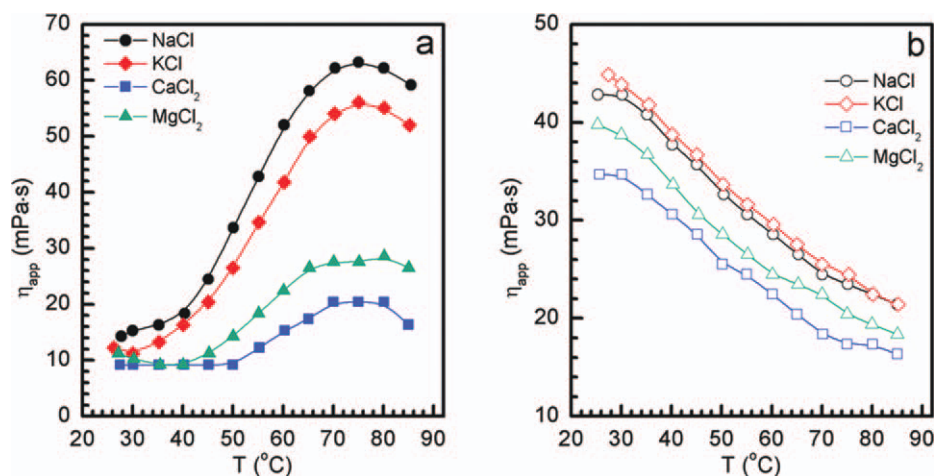
Figure 9 Apparent viscosity plotted as a function of temperature on polymer solution viscosity for (a) PADAS and (b) PAMS in different chlorides brines ($C_p = 0.4\%$, cation concentration = $0.2M$, $\dot{\gamma} = 10$ s^{-1}). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the large cations, and the chains coil up, thus reducing the apparent viscosity of PADAS in $0.2M$ different electrolytes with the same anion, Cl$^-$, aqueous solutions. The electrostatic shield effect of the cation depends strongly on the valency charge of the cation, for example, as compared with divalent Mg^{2+} and Ca^{2+}, the electrostatic shield effect of monovalent Na$^+$ and K$^+$ as counterions for the repulsion between aninons on the polymer, $-SO_3^-$, is weaker.[36,37] It must be pointed out that the thermoviscosifying capability of PADAS is relevant to the change in the polarity of aqueous solution and the electrostatic shield effect of contrary ion for the repulsion between anion on the polymer, SO_3^-. So, the thermoviscosifying ability of PADAS mainly depends on the competition of these two effects. Though thermoassociative water-soluble polymers were widely investigated by Hourdet et al.[27–30], divalent cation effects on the thermoviscosifying ability were not documented. So, considering the target applications in oil industry, we investigated for the first time the effects of divalent cations, such as Ca^{2+} and Mg^{2+}, commonly present in oil reservoir, on the thermoviscosifying ability of thermoassociative water-soluble polymers.

As seen from Figure 9(b), one can find that the viscosity of PAMS aqueous solutions in $0.2M$ different electrolyte solutions with the same anion Cl$^-$ decreases with increasing temperature, and the decrease in the apparent viscosity of polymer solution in the order CaCl$_2$ > MgCl$_2$ > NaCl $\approx$ KCl. From a general point view, the negative charges on the polymeric chains could be effectively neutralized by the large cation, and the electrostatic shield effect of divalent cation is stronger than that of monovalent cation. Thus the viscosity of PAMS solution in

electrolyte solutions of CaCl$_2$ and MgCl$_2$ is much lower than that in the electrolyte solutions of NaCl and KCl in the same condition of temperature.

CONCLUSIONS

Thermoassociative water-soluble polymers are of increasing importance as "smart" materials in a variety of applications, particularly in enhanced oil recovery (EOR) process. However, different inorganic salts, commonly present in the oil reservoirs, will influence the rheological behaviors of polymer solutions for incremental oil recovery. In this work, we compared the effects of salinity, inorganic salt species on the viscosifying behaviors of thermoassociative PADAS and nonassociative PAMS. The PADAS terpolymer exhibits obvious thermoviscosifying behavior in different inorganic salt electrolyte solutions, and the magnitude of thermoviscosifying effect is largely enhanced with increasing salt concentration, whereas the PAMS copolymer shows only monotonous thermothinning property under identical conditions and the viscosity of the polymer solution decreases upon increasing salinity. Moreover, T_{ass} of PADAS can be tuned by changing salt species and salinity of the polymer solution. The thermoviscosifying capability of PADAS is relevant to the change in the polarity of aqueous solution and the electrostatic shield effect of contrary ion for the repulsion between anion on the polymer, SO_3^-, depends mainly on the competition of these two effects. These peculiar thermoviscosifying properties enable PADAS to find potential applications in severe conditions such as high-temperature and high-salinity oil reservoirs.

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