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Characterization and Solution Properties of a Novel Water-soluble Terpolymer For Enhanced Oil Recovery

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Summary

To obtain the polymer with good ageing property and the application in medium- and low-permeability oil layers, the water-soluble hydrophobically acrylamide-modified terpolymer (PAAN) with sodium 2-acrylamido-2-methylpropane sulphonate (NaAMPS) and 2-vinylnaphthalene as a hydrophobic monomer (VN) was synthesized by the micellar copolymerization. The differential scanning calorimetry (DSC) and thermogravimetry (TG) results show that large aromatic groups are incorporated into the PAAN polymer in the form of microblock structure and can increase the rigidity of molecular chains, resulting in the good thermal property of the polymer. The measurement results for intrinsic viscosities of PAAN indicate that the molecular weights of polymers are low and the breakage of the polymer chains can not occur at high shear rate. The polymer exhibits salt-thickening, temperature-thickening, thixotropy and good anti-ageing property. The hydrophobic microdomains and associating three-dimensional networks in the aqueous solution of PAAN were observed respectively by flourescent probe and ESEM.

Introduction

Partially hydrolyzed polyacrylamide (HPAM) is widely applied in enhanced oil recovery (EOR), which role is to increase the viscosity of the aqueous phase to reduce the fluidity ratio of water and oil during EOR processes [1]. The viscosification of the polymer relies on its superhigh molecular weight. For HPAM used as formulated polymer solutions in polymer flooding applications, the molecular weight (*MW*) often ranges from 1.0×10^7 to 2.8×10^7 g mol^{-1} and also, the degree of hydrolysis (*DH*) ranges from 10% to 30%. However, in medium- and low-permeability oil reservoirs (permeability: 0.1~0.5 μm^2), HPAM easily stops up porosities because of big sizes of molecules. High shear rate causes the breakage of the polymer chains, resulting in the irreversible decrease of viscosity, and the viscosity retention ratio is in the range of 20%-30%. Therefore, to obtain a polymeric thickener which sizes of molecular chains can match those of porosities in medium- and low-permeability oil reservoirs with high temperature and salinity, in the paper, we synthesized a novel hydrophobically associating acrylamide-modified terpolymer (PAAN) with sodium 2-acrylamido-2-

methylpropane sulphonate (NaAMPS) and 2-vinylnaphthalene as a hydrophobic monomer (VN) by the micellar polymerization. Hydrophobically associating acrylamide-based copolymers with a small amount of hydrophobic groups have been extensively studied in recent years [2, 3]. But most hydrophobic monomers easily hydrolyze at high temperature [4-6], and for the polymers, the viscosities at low polymer concentration are not high enough to be applied in EOR because of weak interactions of hydrophobic units [7].

In the paper, the molecular block structure and thermal stability of the PAAN polymer were studied by DSC and TG, respectively. The solution viscosities of PAAN polymers were investigated as a function of VN amount, temperature, shear rate and salt concentration. The flourescent probe analysis and Environmental scanning electron microscope (ESEM) were used respectively to observe the nonpolarity of hydrophobic microdomains and true microstructures of PAAN in aqueous and brine solution to reveal hydrophobic associations of VN. We wish that the synthesized PAAN polymers can avoid the inconsistency between low molecular weight and high solution viscosity, and are exactly accordant with expected molecular structure and properties of polymeric oil-displacing agent applied in medium- and low-permeability oil reservoirs with high temperature and salinity.

Experimental

Reagents

AM was recrystallized twice from chloroform. 2-vinylnaphthalene was purchased from Acros Organics Company. Other reagents were analytically pure and used without further purification.

Instrumentation

The UV spectrum was obtained with a UV-240 spectrophotometer. The FT-IR spectrum was conducted with a NICOLET-560 FT-IR spectrophotometer by using KBr disks. The purified PAAN copolymer solution in D_2O was measured by a 400MHz INOVA-400 instrument to obtain the ^{1}HNMR spectrum. Fluorecence spectra were recorded on a Hitachi-850 spectrophotofluorometer at 25 degrees centigrade. The carbon, nitrogen and hydrogen contents of the PANN polymers were determined by CARLO ESRA-1106 elemental analyzer.

The glass transition temperature of PAAN was measured by a NETZSCH DSC204 instrument, and N_2 was used as the experimental atmosphere, and the heating rate is 20 degrees centigrade/min. The thermogravimetric curve of PAAN was obtained by a PE TGA7 thermogravimetric analyzer, and N_2 was used as the experimental atmosphere, and the heating rate is 10 degrees centigrade/min. The apparent viscosities of polymer solutions were measured by a Brookfield DV3 R27112E viscometer at the shear rate of 7.34 s^{-1} at 25 degrees centigrade. The intrinsic viscosities were measured by a 0.6 mm Ubbelohde capillary viscometer at (30.0 ± 0.1) degrees centigrade and 1 mol L^{-1} sodium nitrate is used as a solvent.

The natural aggregating morphology of the hydrophobically associating polymer molecules in aqueous and brine solutions were observed by ESEM XL30. The temperature in the sample room was maintained at -3.5 degrees centigrade and the pressure was controlled below 5 Torr to keep samples under solution state during the whole observation.

Synthesis of Copolymers

The PAAN copolymers were synthesized by the free radical micellar copolymerization [8]. A 100 mL three-necked round flask was equipped with a mechanical stirrer, nitrogen inlet and outlet. AM (5.0 g, 0.0704 mol), AMPS (1.2796 g, 0.006174 mol) and sodium dodecyl sulphate (SDS) (1.0615 g) were dissolved in 25.48 mL of distilled water and the solution was placed in the flask. NaOH was used to control the pH value of the reaction solution between 6 and 7. The mixture was stirred for 15 min, and VN (0.08926 g, 5.7880×10^{-4} mol) was then added into the reaction flask. The flask was purged with N_2 for half an hour. The reactant solution was heated to 50 degrees centigrade with stirring in a tempering kettle under nitrogen atmosphere, and 7.06 mL of 0.005 mol L^{-1} $K_2S_2O_8$ solution was then added into the reactant solution. After the polymerization proceeded for 16 h at 50 degrees centigrade, the reaction mixture was diluted with five volumes of distilled water, and two volumes of acetone was then added with stirring to precipitate the polymers. The polymers were washed with acetone twice and extracted with ethanol by the Soxhlet extraction instrument for two days to remove all traces of water, surfactant, residual monomers and initiator. Finally, the polymers were dried under *vacuo* at 50 degrees centigrade for three days.

In the absence of VN, the NaAMPS/AM copolymer (PAA) was synthesized, and the reaction conditions and purification method were the same as mentioned earlier.

For the UV spectrum of PAAN, there is a typic absorption peak at 237 nm attributed to the aromatic polyring. FT-IR of PAAN (cm^{-1}): -N-H stretch, 3416.70; C=O stretch, 1662.76; $-CH_3$, $-CH_2$, -CH stretch, 2851.92, 2932.82, 2785.19; $-CH_3$, $-CH_2$, -CH bending, 1451.79, 1414.81, 1345.96; =C-H in phenyl stretch, 3207.41; =C-H in phenyl bending, 761.59; $-SO_3^-$ 1188.14, 1119.87, 1040.45. ^{1}H- NMR (400MHz) shifts δ (ppm): 4H (-CH of phenyl), 7.464-7.965; 2H ($-CH_2$ of VN main-chain), 3.458; 1H (-CH of VN main-chain), 3.224; 6H ($-CH_3$ of NaAMPS side chain), 1.520; 2H ($-CH_2$ of AMPS side chain), 3.687; 2H ($-CH_2$ of NaAMPS main chain), 1.787; 1H (-CH of AMPS main chain), 2.369; 2H ($-CH_2$ of AM main chain), 1.820; 1H (-CH of AM main chain), 2.60.

Results and Discussion

Differential Scanning Calorimetry and Thermogravimetric Analysis

Figure 1 shows the molecular structure of PAAN, which is determined by ^{1}HNMR spectrum. The chain structure of PAAN was successfully characterized by the differential scanning calorimetry (DSC). However, the block structure of hydrophobic segments was previously determined by flourescent probe [9]. Figure 2 shows the

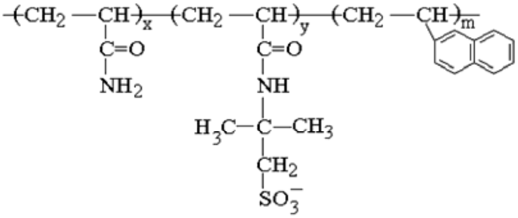


Figure 1. Schematic representation of molecular structure of PAAN

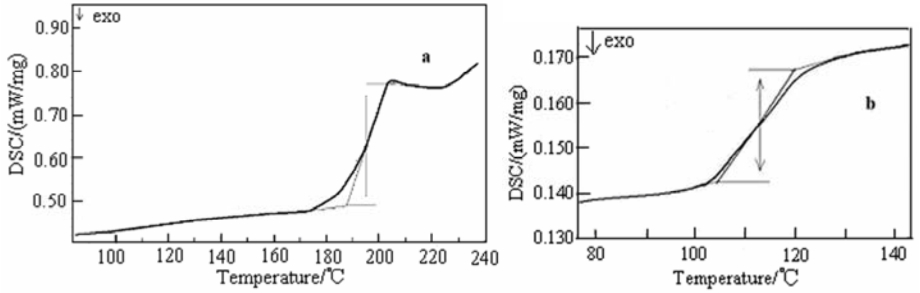


Figure 2. Glass transition temperature of PAAN. (a) heating rate: 20 degrees centigrade/min. (b) enlarged graph of (a) in the range of 80-140 degrees centigrade

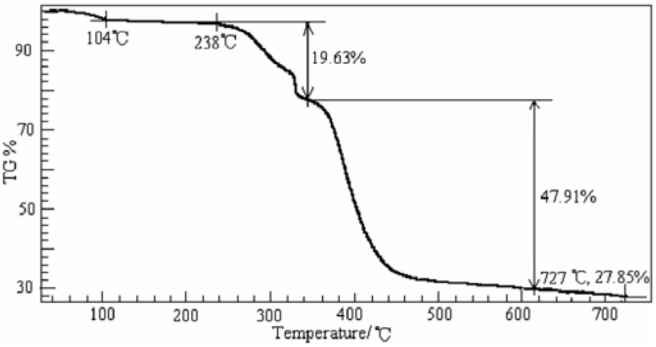


Figure 3. TG analysis curve of PAAN

glass transition temperature (T_g) of PAAN, and Figure 2 (a) is enlarged in the range of 80-140 degrees centigrade to obtain Figure 2 (b). T_g of PAAN includes 196 degrees centigrade of random AM/NaAMPS hydrophilic segments and 112 degrees centigrade of microblock VN hydrophobic segments. The result indicates that VN units are incorporated into PAAN chains to form microblock structures which favor intermolecular hydrophobic associations. T_g (196 degrees centigrade) of hydrophilic segments in PAAN is higher than that (163 degrees centigrade) of homopoly-acrylamide, which suggests that the rigidities of polymer chains increase because of the steric effect of aromatic ring in VN units and the polarity of NaAMPS units.

Figure 3 displays the heat-resistant property of PAAN. The loss of weight (2.1%) from 20 degrees centigrade to 104 degrees centigrade is resulted in by volatilization of water molecules in the polymer; and the weight is constant from 104 degrees centigrade to 238 degrees centigrade, and the polymer begins to decompose at 238 degrees centigrade. The result indicates that the initial decomposition temperature (238 degrees centigrade) of the polymer is obviously higher than that (210 degrees centigrade) of homopolyacrylamide [10], and the thermal stability of the polymer is better than homopolyacrylamide, attributable to the incorporation of rigid aromatic polyring and big polar groups of NaAMPS units into the polymer.

Composition and Intrinsic Viscosity

The polymerization conditions and composition are shown in Table 1 and Table 2, respectively. The reaction temperature of all samples is 50 degrees centigrade, and the pH value of reaction solution is 6~7. As shown in Table 2, with increasing VN feed amount, the intrinsic viscosity of polymer decreases because of steric obstruction of the hydrophobic monomer, indicating that the molecular weight of polymer reduces. The intrinsic viscosities of all polymers are low, indicating that the molecular sizes of the polymers can match the sizes of porosities in medium- and low-permeability oil layers [11]. For the PAAN polymers, the intrinsic viscosity ([η]) of 4.21-5.74 dL g^{-1} is about equal to the *MW* of 5.56×10^5-8.90×10^5 according to the equation $[\eta] = 6.8\times10^{-4} M^{0.66}$. In polymer flooding applications, the ratio R_h / R_g value of pore throat radius (R_h) to gyration radius (R_g) of polymer coil must be higher than 5. For HPAM with the *MW* of 6.5×10^6 (R_g = 0.27 μm) and the core with the permeability of 0.085 μm^2 (R_h = 1.25 μm), the R_h / R_g value is 4.63. Therefore, For the PAAN polymers and the cores with the permeability of 0.1~0.5 μm^2, the R_h / R_g value is absolutely higher than 5. The molar compositions of polymers are higher than the corresponding monomer feed compositions for VN because of the local concentration effect of VN monomer solubilized within the surfactant micelles.

Table 1. Polymerization conditions of PAAN polymers

Sample	Reaction conditions			
	M1: M2 : M3[a]	SDS[b] (wt %)	Monomers[c] (wt %)	$K_2S_2O_8$[d] (wt %)
PAAN1	91.6 : 8.0 : 0.4	4.0	20	0.15
PAAN2	91.4 : 8.0 : 0.6	4.0	20	0.15
PAAN3	91.25 : 8.0 : 0.75	4.0	20	0.15
PAAN4	91.0 : 8.0 : 1.0	4.0	20	0.15
PAA	92.0: 8.0: 0	0	20	0.15

[a] M1, acrylamide; M2, NaAMPS; M3, VN feed molar compositions.
[b] SDS =sodium dodecyl sulphate, mass percent composition in water.
[c] Monomers=total monomer concentration, mass percent composition in water.
[d] Mass percentage relative to total monomer.

Table 2. Compositions and properties of PAAN polymers

Sample	Elemental composition				Molar composition M1: M2 : M3[a]	Intrinsic viscosity(dL g^{-1})
	C (wt%)	*N* (wt%)	*H* (wt%)	*S* (wt%)		
PAAN1	48.60	16.21	6.79	3.99	88.75: 10.70: 0.55	5.74
PAAN2	48.86	16.32	6.80	3.79	89.20: 10.09: 0.71	4.95
PAAN3	49.08	16.31	6.81	3.70	89.23: 9.84: 0.93	4.21
PAAN4	49.41	16.36	6.82	3.53	89.48: 9.32: 1.20	3.80
PAA	48.02	14.34	6.77	4.24	88.54: 11.46: 0	7.02

[a] M1, acrylamide; M2, NaAMPS; M3, VN feed molar compositions.

Effect of VN amount on Solution Viscosity

Figure 4 shows the influence of VN feed amount on apparent viscosity of PAAN in aqueous solution. For PAAN3 with 0.75 mol% VN, the solution viscosity increases dramatically with increasing polymer concentration above 0.05 g dL^{-1}, which is the critical association concentration (C_p*). With increasing the VN feed amount from

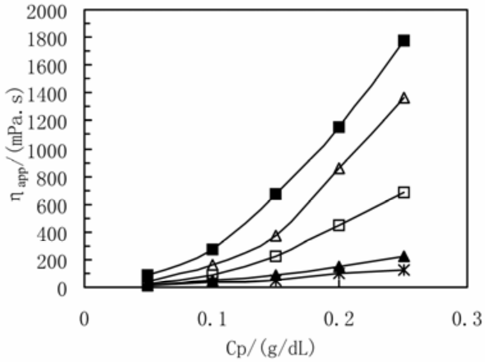


Figure 4. Influence of VN feed amount on apparent viscosity of PAAN aqueous solution. ▲ 0 mol%; □ 0.4 mol%; △ 0.6 mol%; ■ 0.75 mol%; ✳ 1.0 mol%

0.4 mol% to 0.75 mol%, the thickening ability of PAAN is strengthened, and For PAAN4 with 1.0 mol% VN, the solution viscosity is the lowest. For comparison, the concentration dependence of the solution viscosity for PAA is also shown in Figure 4, but the PAA polymer only displays a linear relationship of solution viscosity versus polymer concentration. The results show that despite low molecular weight of the PAAN polymers the incorporation of small amounts of aromatic groups into the polymers leads to the strong intermolecular hydrophobic associations and the high viscosity values of polymer solutions at a low polymer concentration. For HPAM with the same intrinsic viscosity of 4.21-5.74 dL $g^{-1}$and the degree of hydrolysis of 25%, the solution viscosity is greatly low and is only 7-10 mPa.s in 0.684 mol L^{-1} NaCl at the polymer concentration of 0.5 g dL^{-1}, and thus, the HPAM polymer is impossibly applied in EOR because the polymer concentration should be below 0.2 g dL^{-1}.

Effect of NaCl on Solution Viscosity

Figure 5 shows the influences of NaCl on solution viscosities of the PAAN and PAA Polymers. With increasing NaCl concentration, the solution viscosity of 0.2 g dL^{-1} PAAN3 first decreases, and then increases, and finally decreases. However, the PAA polymer behaves the conventional polyelectrolyte behavior because of the shielding of charges in the brine solution. By addition of a small amount of NaCl in the polymer

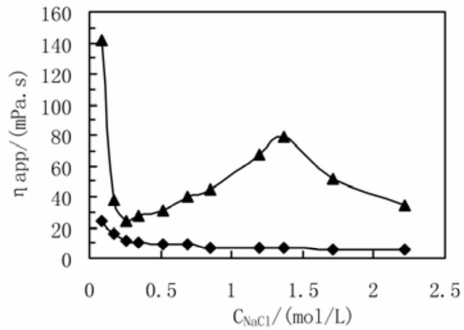


Figure 5. Effect of NaCl concentration on apparent viscosities of polymer brine solution. Polymer concentration: 0.2 g dL^{-1}. ▲ PAAN3; ♦ PAA

solution, charge shielding effect of Na^+ on $–SO_3^-$ along the polymer chain results in coiled molecules, and intermolecular hydrophobic associations are weakened. With the increase of NaCl concentration, the solution polarity induced by electrolytes increases, resulting in reinforced intermolecular associations of the hydrophobic groups and the good salt-thickening behavior of the polymer solution. However, with further increasing the NaCl concentration, the hydrophobic microstructures turn more compact, and finally condensed aggregates associate with each other to form larger aggregates, resulting in the phase separation and the decrease of the apparent viscosity.

Effect of Temperature on Solution Viscosity

Figure 6 shows influence of temperature on apparent viscosities of polymer brine solution. The solution viscositiy increases with increasing temperature from 20 degrees centigrade to 40 degrees centigrade, and then decrease above 40 degrees centigrade. Compared with PAAN3, with increasing temperature, the solution viscosity of PAA only decreases because of faster movement of polymer chains and weaker hydrogen bonding. The results indicate that the increase of temperature is favourable to intermolecular associations in a certain range of temperature. This is due to the endothermic process of entropy increase for hydrophobic association at a low temperature range [12].

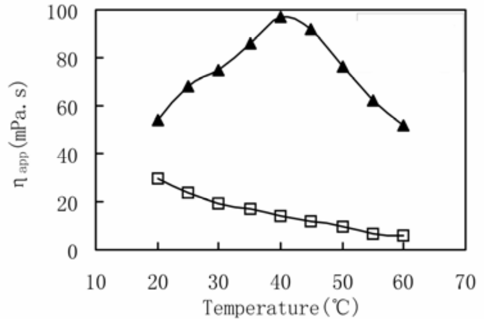


Figure 6. Influence of temperature on apparent viscosities of polymer brine solution. Polymer concentration of PAAN3 and PAA: 0.2 g dL^{-1}; ▲ PAAN3, NaCl concentration: 1.19 mol L^{-1}; □ PAA, NaCl concentration: 0.0855 mol L^{-1}

Effect of Shear Rate on Solution Viscosity

Figure 7 shows the viscosity versus shear rate for PAAN3 in brine solution at 25 degrees centigrade. With increasing shear rate, the viscosity first increases below 6 s^{-1}, and then reduces sharply, and Finally changes slightly because of the balance between intermolecular association and disassociation, which exhibits pseudoplastic behaviour. Upon gradually reducing shear rate, the viscosity is lower slightly than the primary viscosity at a given high shear rate and does not recover immediately, indicating that the intermolecular hydrophobic associations take place at a certain rate. At a low shear rate range, the shear-thickening behavior is attributable to the expansion of polymer chains and the transition of intramolecular associations to intermolecular associations upon shear. The results imply that the intermolecular associations are reversible, and

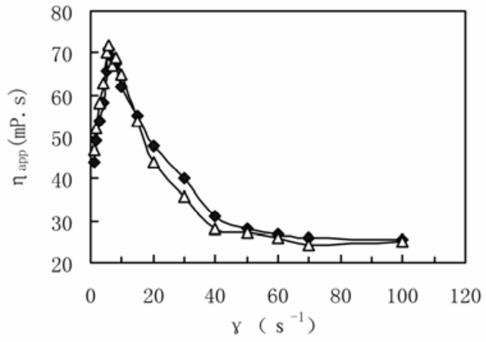


Figure 7. Influence of shear rate on apparent viscosity of 0.2 g dL^{-1} PAAN3 in 1.19 mol L^{-1} NaCl solution. △ shear rate decrease, ♦ shear rate decrease

all aggregates break at high shear rate. In addition, the results also show that the polymer does not degrade and the molecular structure is stable upon shear. Therefore, the PAAN polymer with low molecular weight can be applied in medium- and low-permeability oil layers [11], and the polymer chains can pass through easily pore throats with small apertures. After the polymer solution flows through pore throats, the polymer chains associate with each other to increase solution viscosity via molecular interactions of hydrophobic groups in pores because of lower shear rate, resulting in reducing the fluidity ratio of water to oil and enhancing oil recovery.

Ageing Effect

Table 3 shows the ageing property of PAAN3 in 1.19 mol L^{-1} NaCl solution with saturated oxygen at 80 degrees centigrade. After ageing for 90 days at 80 degrees centigrade, the viscosity retention ratio of PAAN3 brine solution is 88.3%, which is higher than that (45.6%) of 0.1 g dL^{-1} HPAM (M: 2.1×10^7, HD:25-30%) in 0.130 mol L^{-1} NaCl solution after ageing for 90 days at 34 degrees centigrade under vacuum [13]. The result shows that the polymer has good anti-ageing property in brine solution at 80 degrees centigrade because of the incorporation of VN into copolymer chains. Aromatic polyrings in VN can increase rigidities of polymer chains, and can effectively interfere with degradation of PAAN and the hydrolysis of amido groups in the polymers. The ageing result is consistent with the DSC and TG results.

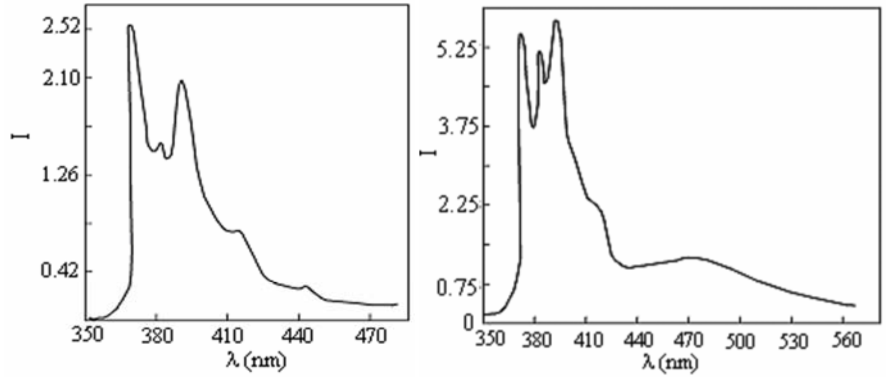
Table 3. Apparent viscosity of PAAN3 in brine solution after ageing

	Ageing time/d									
	0	1	5	10	15	30	45	60	70	90
Viscosity/ (mPa.s)	68.0	73.0	77.2	81.6	82.0	75.8	71.9	67.2	64.3	60.0

Polymer concentration: 0.2 g dL^{-1}, NaCl concentration: 1.19 mol L^{-1}.

Hydrophobic Microdomains and Associating Microstructure of PAAN

Figures 8 shows the pyrene emission spectrum in water and 0.05 mol L^{-1} SDS aqueous solution, respectively. Pyrene is widely used as a fluorescent probe as among the various fluorescent probes, pyrene is most sensitive to the change in the vibrational



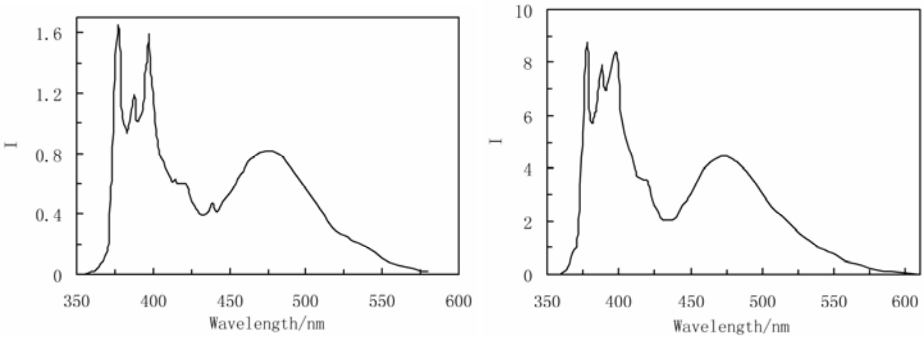
(a) Water **(b)** 0.05 mol L^{-1} SDS aqueous solution

Figure 8. The pyrene emission spectrum in water and SDS aqueous solution

fine structure of its emission spectrum [9, 14]. The intensity ratio I_1/I_3 of the first (375 nm) to the third (385 nm) vibronic peaks in the fluorescence emission spectrum of pyrene is sensitive to the polarity of the local microenvironment of the pyrene probe. Thus, the formation of hydrophobic microdomains in aqueous solution can be evidenced by a decrease of I_1/I_3 ratio. As shown in Figures 8, the I_1/I_3 value decreases from 1.76 in water to 1.05 in 0.05 mol L^{-1} SDS solution where the pyrene probe is located in the micellar aggregates.

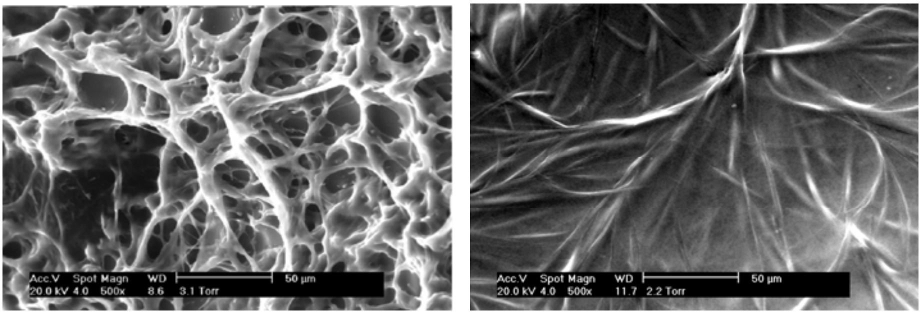
Excimer formation occurs when the concentration of pyrene in the hydrophobic microenvironment is high enough for an excited pyrene (Py^*) and a pyrene in its ground state to come into close proximity during the Py^* lifetime. Pyrene excimer emission is characterized by a broad featureless spectral emission peak centered at 475nm. Hence, the intensity ratio I_e/I_m of the excimer emission peak to the monomer peak (399 nm) has often been used as an indicator of the degree of interaction between fluorophores. For hydrophobically associating polymer, the I_e/I_m ratio provides a measure to characterize the association degree of hydrophobic segments on the polymer chains.

Figures 9 show the pyrene emission spectrum for 0.2 g dL^{-1} PAAN3 in aqueous and 1.19 mol L^{-1} NaCl solutions, respectively. The I_1/I_3 values of the aqueous and brne



(a) Aqueous solution **(b)** 1.19 mol L^{-1} NaCl solution

Figure 9. Fluorescence spectra of pyrene in PAAN solutions. Polymer concentration: 0.2 g dL^{-1}



(a) Aqueous solution **(b)** 1.19 mol L^{-1} NaCl solution

Figure 10. ESEM image of 0.2 g dL^{-1} PAAN3 in aqueous and brine solution

solutions are 1.38 and 1.10, respectively. The results shows that hydrophobic microdomains are formed in aqueous and brine solution of the polymer, and the I_1/I_3 value in brine solution is lower than that in aqueous solution, which indicates that the hydrophobic domains become more compact by addition of NaCl, leading to the increase in non-polarity of pyrene environments. The I_e/I_m values of the aqueous and brine solutions are 0.50 and 0.53, respectively. The result further suggests that intermolecular hydrophobic association in brine solution is stronger than that in the aqueous solution, and by addition of NaCl, the solubility of pyrene in hydrophobic domains increase.

The natural hydrophobically associating morphology of PAAN in aqueous solution was observed by ESEM, and the morphology is shown in Figure 10 (a). Despite low molecular weight the polymer chains aggregate together to form three-dimensional networks via strong intermolecular hydrophobic associations, resulting in the high solution viscosity, and the diameters of the networks bones are 1-6 µm. As shown in Figure 10 (b), for 0.2 g dL^{-1} PAAN3 in 1.19 mol L^{-1} NaCl aqueous solution, the continuous network structures in aqueous solution are destroyed by addition of salt because of electrostatic shielding of Na^+ on $-SO_3^-$ groups along the polymer chains, and lots of compact dendritic aggregates with different sizes (diameter: 0.5-4 µm) are formed, resulting in the decrease in solution viscosity. The fluorescent probe and ESEM results reveal that the thickening ability of the polymers is mainly dependent on intermolecular hydrophobic associations.

Conclusions

The hydrophobically acrylamide-modified terpolymers (PAAN) with sodium 2-acrylamido-2-methylpropane sulphonate and the hydrophobic monomer VN were synthesized by the micellar copolymerization technique. The DSC result indicates that hydrophobic block structures of PAAN are formed and the incorporation of VN into the polymer enhances the rigidity and thermal stability of molecules. The molecular chains of PAAN can not break at high shear rate because of the low molecular weight, and the feed amount of VN affects remarkably the apparent viscosity of PAAN solution because of strong hydrophobic interaction. Despite the low molecular weight the PAAN polymer exhibits temperature-thickening, thixotropy, pseudoplastic behavior, anti-shearing and the good salt-thickening behavior in brine solution below 1.367 mol L^{-1} NaCl. The PAAN polymer with rigid aromatic polyring displays good anti-ageing property in brine solution at 80 degrees centigrade which is consistent with the DSC and TG results. The physical three-dimensional networks are formed in the aqueous solution of 0.2 g dL^{-1} PAAN because of strong hydrophobic associations, leading to good solution viscosities. Our assumptions are demonstrated to be valid by the above mentioned results.

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