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ORIGINAL CONTRIBUTION

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Aggregation behavior of acrylamide/2-phenoxyethyl acrylate and its interaction with sodium dodecyl sulfate in aqueous solution studied by proton 1D and 2D NMR

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Abstract ^{1}H NMR self-diffusion coefficient, spin-lattice relaxation time, spin-spin relaxation time, and two-dimensional nuclear Overhauser enhancement (2D NOESY) measurements have been used to study the association behavior of a novel hydrophobically associating copolymer composed of acrylamide (AM) and a small amount of 2-phenoxyethyl acrylate (POEA), and its interaction with the anionic surfactant sodium dodecyl sulfate (SDS). Three sets of copolymers with approximately the same composition but with different hydrophobic POEA contents were investigated. The POEA contents for these copolymers were about 1.41, 1.03, and 0.56 mol% respectively, as validated by ^{1}H NMR spectra. Self-diffusion coefficient measurements show that the aggregation process occurs in a relative narrow concentration range and the critical association concentrations (*cac*s), of these copolymers are within this narrow concentration range, which are in agreement with those measured by viscosity measurements (6 g L^{-1}). Above this concentration, the hydrophobic POEA moieties are found to associate and possibly build a transitory three-dimensional network along the polyacrylamide (PAM) backbones, which induces a strong decrease in NMR parameters including self-diffusion coefficients and relaxation times. The surfactant SDS showed a significant interaction with the copolymer in the dilute solution. Addition of SDS resulted in the binding of SDS on copolymer POEA-PAM segments and reinforced the interchain transient network formation of copolymer at a concentration below its *cac*. In the SDS/POEA-PAM mixed systems, the hydrophobic methylene groups of the SDS molecules were preferentially located in the vicinity of the phenoxy groups of the POEA hydrophobes.

Keywords Nuclear magnetic resonance spectroscopy · Nuclear Overhauser enhancement · Hydrophobically associating copolymer · Critical association concentration · Sodium dodecyl sulfate

Introduction

Block copolymers have been the subject of intense experimental and theoretical interest for more than two decades because their special chemical structure yields unusual physical properties. Block copolymers are applied in various fields, as surfactants, adhesives, thermoplastics, and thermoplastic elastomers [1, 2].

Recently there has been considerable interest in a relatively new class of polymers containing small numbers of hydrophobic substituents. These so-called hydrophobically modified water-soluble polymers are based on a hydrophilic backbone, such as polyacrylamide (PAM) [3, 4] or poly(sodium acrylate) [5] have become the subject of extensive research because of their use as aqueous viscosity modifiers in oil recovery and latex paint systems. The incorporation of a few hydrophobic groups results in systems with unique rheological characteristics owing to their striking thickening efficiency in aqueous solution, as the hydrophobic moieties form interchain hydrophobic aggregates above a certain concentration (*cac*). The *cac*s of block copolymers have been investigated using many different techniques including nuclear magnetic resonance (NMR) [6, 7], differential scanning calorimetry (DSC) [8], static and dynamic light scattering [7, 9] and fluorescence spectroscopy [10], and small-angle neutron-scattering (SANS) [11]. The results show that the *cac* is not a definite concentration but more a concentration range.

The association between polymers and surfactants in aqueous solutions has attracted much interest recently [12]. Studies show that ionic and even zwitterionic surfactants interact strongly with copolymers [13, 14]. Most work has focused on the linear copolymer, and a smaller amount on the hydrophobically modified copolymers. The properties of aqueous solutions of hydrophobically modified water-soluble copolymers can be improved by the addition of surfactants, and this behavior is of fundamental importance for many industrial applications [15]. The anionic sodium dodecyl sulfate (SDS) is the most frequently used surfactant [16, 17]. In some works, the cationic cetyl trimethyl ammonium bromide (CTAB) [18], or nonionic surfactant *t*-octylphenoxy polyoxyethanol (TX-100) [19] were used. The interactions in aqueous solution of SDS with copolymers of hydrophobically modified PAM have been examined [20]: A strong increase in viscosity was observed upon the addition of SDS below its critical micelle concentration, and the complex rheological behavior observed is explained in terms of the balance between inter and intrachain liaisons.

Nuclear magnetic resonance (NMR), in paricular the 2D NMR technique has proved a very powerful method for studying the detailed structure of the micelles and for posing and answering many questions regarding the microenvironments encountered in the interfacial systems of other kinds, such as membranes [21]. The critical association concentration (*cac*) of a non-ionic polymer surfactant was determined by the ^{1}H NMR self-diffusion method [22]. Recently, we studied the association behaviors of dendritic polyether-modified PAM with SDS in aqueous solution by ^{1}H NMR relaxation and self-diffusion measurement [23]. In this work, the aim is elucidation of the aggregation process and detailed structure of a novel, hydrophobically associating copolymer composed of acrylamide (AM) and a small amount of 2-phenoxyethyl acrylate (POEA) in aqueous solution and its interaction with the anionic surfactant SDS, via study of its self-diffusion coefficient, relaxation times and two-dimensional nuclear Overhauser enhancement (2D NOESY) at different concentrations. A possible mechanism for such interactions is proposed.

Materials and methods

Three sets of hydrophobically associating copolymers composed of AM and different amounts of POEA were synthesized by the Technical Institute of Physics and Chemistry, of the Chinese Academy of Sciences [24], and used without further purification. M_w was about 8×10^6 measured by the viscosity method [25]. SDSwas from Across Organics, USA. The D_2O (99.5% d) was obtained from the Beijing Chemical Plant of China. The concentrations of the POEA-PAM samples in D_2O solutions used in NMR experiments ranged from 1 g L^{-1} to 15 g L^{-1}. A series of different compositions of SDS/POEA-PAM solutions were made up and kept 1 week prior to the experiments. NMR experiments were performed at 298 K on a Varian INOVA-500 NMR spectrometer with a ^{1}H frequency of 500.13 MHz. Inversion recovery and Carr-Purcell-Meiboom-Gill (CPMG) pulse sequences were used for T_1 (spin-lattice) and T_2 (spin-spin) relaxation time measurements, respectively. Self-diffusion coefficients were measured by the longitudinal eddy-current delay with bipolar pulse pair (LED-BPP) pulse sequence [26]. D_2O was used as solvent instead of water in order to weaken the water signal. Meanwhile, the presaturation method was used to further suppress the proton signal of solvent. 2D NOESY experiments were performed with the standard three-pulse sequence [27]. Thirty-two accumulations, $t_2\times t_1 = 2k\times256$ sampling data points array, and mixing times of 80 ms were used. The data point array $F_2\times F_1 = 2k\times512$ was used in the Fourier transformation after the zero filling.

Results and discussion

Copolymer composition

The formula and proton numberings of POEA-PAM are shown in Fig. 1. ^{1}H NMR spectrum of POEA-PAM copolymer for polymer 1 with a concentration of 3 g L^{-1} is shown in Fig. 2. The POEA hydrophobic contents can be determined by comparing the peak integral of the meta-phenoxy proton, P8, (at $\delta = 7.48$–7.35 in D_2O) of the copolymer to that of the methine protons, P2 plus P4, of the backbone (at $\delta = 2.48$–2.08 in D_2O) and of the

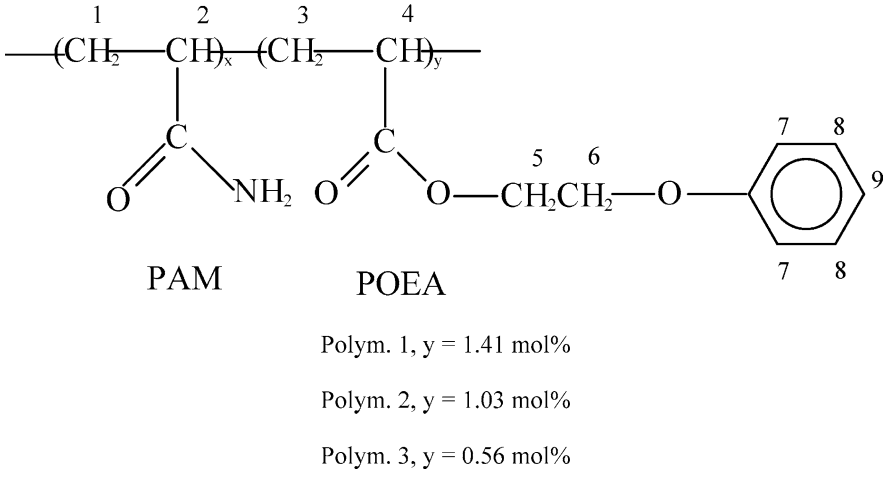

Fig. 1 The formula and proton numberings of three sets of 2-phenoxyethyl acrylate (*POEA*)–polyacrylamide (*PAM*) copolymers

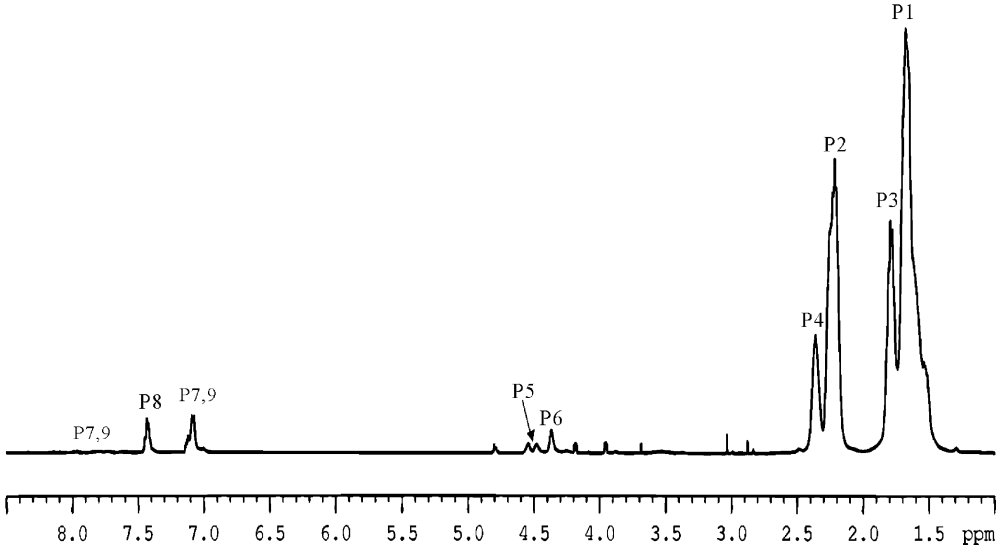

Fig. 2 ^{1}H NMR spectra of POEA-PAM with a POEA content of 1.41 mol% at 3 g L^{-1}

methylene protons, P1 plus P3, of the backbone (at $\delta = 1.98$–1.48 in D_2O). Quantitative polymerization of the POEA blocks was estimated at about 1.41 mol% for the polymer 1. By the same means, the POEA contents of polymers 2 and 3 can be easily estimated at 1.03 and 0.56%, respectively. The results are in good agreement with those measured by UV absorption spectroscopy [24].

Critical association concentration

In principle, one can use data on any physical property that depends on the particle size or the number of particles to determine the *cac*. Most frequently, breaks or discontinuities in plots of such properties as the viscosity, surface tension, or light scattering as a function of concentration have been used for this purpose. The *cac* can also be determined from the acute change in the self-diffusion coefficient as a function of concentration. It is based on the large intrinsic difference in self-diffusion rates for monomeric and associated molecules. The NMR self-diffusion coefficient measurement method has been widely used to measure the *cac* of various surfactants such as tetra-ethylene *n*-octyl ether [28]. In Fig. 3 is shown the dependence of the self-diffusion coefficient (*D*) on the concentrations for the three sets of POEA-PAM copolymers in D_2O solutions, respectively. It should be emphasized that the self-diffusion coefficients of all proton peaks for each polymer are identical within experimental error, so the *D* values shown in Fig. 3 are the averages of all the peaks for a given polymer.

Obviously, the self-diffusion coefficients of the three sets of copolymers stayed nearly constant at concentrations in the range 1–4 g L^{-1}, followed by a sharp turning point between 4 and 7 g L^{-1} on the concentration dependence of the POEA-PAM aqueous solution curve, as shown in Fig. 3. The descent continued up to a

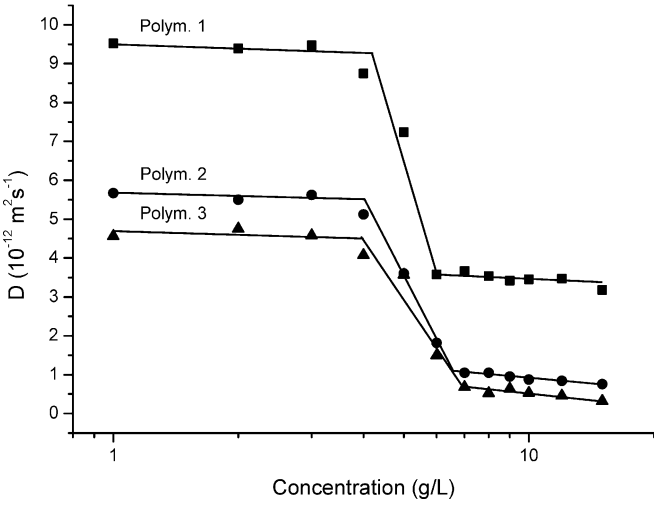

Fig. 3 The self-diffusion coefficients of three sets of POEA-PAM with varying concentrations

concentration of about 7 g L^{-1} and on a further increase in the concentration the self-diffusion coefficients continued to decrease, but slowly. This indicates that the aggregation process of these copolymers occurs in a rather narrow concentration range and the *cac*s of these copolymers are in this narrow concentration range of 4–7 g L^{-1}, which is in agreement with those measured by viscosity measurements (around 6 g L^{-1}) [24]. In contrast to the case of polymer 1, which has more hydrophobic POEA, the self-diffusion coefficients of polymers 2 and 3 were somewhat shorter. This suggests that the less the hydrophobic POEA content, the more seriously the motion is restricted.

Dynamics of POEA-PAM aggregation in aqueous solution

The spin-lattice relaxation times (T_1), spin-spin relaxation times (T_2), and the ratio T_R (T_2/T_1) of the main protons of copolymer POEA-PAM in aqueous solutions at various concentrations below and well above their *cac* for different polymers are shown in Table 1. According to the relaxation rate dependence on the correlation time of the molecule, a deviation of T_2/T_1 from unity shows that the motion is restricted. It is evident that T_2 values of all the methylene and methine protons of POEA-PAM backbone for different polymers are only several tens of milliseconds—very short—at all concentrations studied, and the T_R values of all these proton signals are less than 0.1. This suggests that the motions of POEA-PAM main chain protons deviate from the extreme narrowing condition, and the backbones are densely packed. Motions of these protons are seriously restricted. It is worth noting that T_2 values of the hydrophobic POEA side-chain protons, especially for the phenoxy protons, above its *cac* are markedly shorter than those of the dilute systems, which indicates that the motion of POEA-PAM molecules is more restricted in more concentrated POEA-PAM copolymer systems. Besides, this obvious change of the phenoxy proton relaxation times indicates that the aggregation occurs mostly on the hydrophobic phenoxy moieties. The viscosity values and self-diffusion coefficients of POEA-PAM in the solutions also shown in Table 1 support the existence of interaction among the molecules, i.e., the self-aggregation. Compared to the T_2 values of the protons within the molecules we see that the protons of the main chain are most restricted and the farther the protons are from main chains, the less the motion is restricted.

To obtain useful information regarding the temperature stability of the aggregation, the spin-spin relaxation time (T_2) and the self-diffusion coefficients of copolymer solutions at below and above the *cac* were determined at three different temperatures. The results are displayed in Table 2. We noticed that these data for dilute solutions increased relatively faster than those for concentrated solutions. The fact that the relaxation times and self-diffusion coefficients of concentrated

Table 1 ^{1}H spin-lattice relaxation time (T_1), spin-spin relaxation time (T_2),T_R (T_2/T_1), viscosity (η) and self-diffusion coefficients (D) of $(POEA)_y$-PAM for different polymers in aqueous solution at different concentrations at 298 K. Experimental error < 10%

Polymer	C	Ph			P6			CH (P2,4)			CH_2(P1,3)			η	$D\times10^{12}$
		T_1	T_2	T_R	T_1	T_2	T_R	T_1	T_2	T_R	T_1	T_2	T_R		
	(g/)L	(ms)	(ms)		(ms)	(ms)		(ms)	(ms)		(ms)	(ms)		(mPa s)	(m^2 s^{-1})
1	2	2,155	939	0.44	517	117	0.23	1,006	113	0.11	577	51	0.09	2	9.4
	5	2,082	856	0.41	513	109	0.21	1,001	110	0.11	575	50	0.09	4.2	7.2
	12	1,854	576	0.31	497	95	0.19	1,023	97	0.09	574	47	0.08	15	3.5
2	2	1,992	709	0.35	523	135	0.26	1,008	110	0.11	576	79	0.14	10	5.5
	5	1,956	648	0.33	515	126	0.24	992	96	0.10	574	73	0.13	60	3.6
	12	1,847	546	0.29	496	109	0.22	1,012	83	0.08	557	59	0.10	1500	0.8
3	2	1,826	625	0.34	516	159	0.31	1,037	102	0.10	562	74	0.13	12	4.8
	5	1,809	600	0.33	508	140	0.27	997	90	0.09	556	69	0.12	80	3.6
	12	1,912	498	0.26	510	115	0.22	1,026	79	0.08	569	60	0.11	1350	0.5

Table 2 ^{1}H spin-spin relaxation time (T_2), and self-diffusion coefficients (D) of POEA-PAM in aqueous solution with concentrations below and above their *cac* at alterative temperatures from 298 to 308 K. Experimental error < 10%

Polymer	C (g/L)	T_2 (Ph) (ms)			T_2 (P6) (ms)			T_2 (CH, P2,4) (ms)			T_2 (CH_2, P1,3) (ms)			$D\times10^{12}$ (m^2 s^{-1})		
		298 K	303 K	308 K	298 K	303 K	308 K	298 K	303 K	308 K	298 K	303 K	308 K	298 K	303 K	308 K
1	2	939	1,028	1,207	117	128	157	113	138	159	51	59	68	9.4	54	121
	12	576	597	635	95	99	106	97	106	117	47	53	59	3.5	19	35
2	2	709	729	764	135	154	172	110	117	128	79	87	93	5.5	7.1	8.9
	12	546	552	566	109	115	124	83	86	92	59	65	76	0.8	1.5	2.4
3	2	625	640	667	159	174	192	102	113	119	74	84	92	4.8	5.3	6.2
	12	498	500	504	115	121	127	79	84	89	60	67	76	0.5	0.8	1.2

solutions increased linearly with the increase in temperature studied to different extents indicates that the aggregates are stable at the temperatures studied. We also see that the data for polymers with more hydrophobic POEA parts increased somewhat faster than those of polymers with less POEA parts. This implies that the smaller the POEA content, the more stable the polymer aggregates.

Microstructure of POEA-PAM aggregates in aqueous solution

It is well known that 2D NOESY is an effective method of studying the detailed structure of macromolecules. It gives information about the distance in space between pairs of coupled protons. 2D NOESY contour plots of POEA-PAM-block hydrophobically associating copolymer systems with a mixing time of 80 ms at various concentrations for polymer 1 are shown in Fig. 4, which gives an insight into the interaction of the POEA-PAM molecules and a detailed structure of the aggregates in aqueous solution. We can observe from Fig. 4A–C that the number of cross-peaks increases remarkably with increasing concentration of POEA-PAM from 3 g L^{-1} to 12 g L^{-1}. Only intramolecular cross-peaks between protons (P1-P2, P1-P3, P2-P3, P2-P4, and P3-P4) appear in the 2D map of the dilute POEA-PAM copolymer system (Fig. 4A, 3 g L^{-1}), while cross-peaks between protons of phenoxy groups and those of the main chains of POEA-PAM can be detected in the 2D NOESY maps of the concentrated systems (Fig. 4B–C). It suggests that the distances of these proton pairs are at least no longer than 5 Å, which can be an indication of aggregation. This strongly supports the results discussed above. The appearance of cross-peaks among phenoxy protons, and between phenoxy protons and main chain protons in the 2D maps are an indication that the hydrophobic phenoxy moieties interact and are possibly located in the vicinity of PAM backbones in POEA-PAM aggregates.

Combining the results of self-diffusion coefficients, relaxation times, and 2D NOESY experiments, it seems reasonable to conclude that aggregates form when the polymer concentration is above the *cac*. This aggregation occurs mostly on the hydrophobic phenoxy moieties, which interact and are possibly located near the PAM backbones in POEA-PAM aggregates. The lower the hydrophobic POEA content, the more the motion is restricted and the more stable are the aggregates.

Interaction between POEA-PAM and surfactant SDS in aqueous solution

Molecular dynamics and interactions between POEA-PAM and SDS in the mixed solutions were estimated by the spin-spin relaxation times (T_2), the ratio T_R (T_2/T_1), and the self-diffusion coefficients of the protons of copolymer POEA-PAM and SDS in their mixed aqueous systems at various concentrations below and above their *cac* for polymer 1. The results are shown in Table 3. It is obvious that the T_2 and self-diffusion coefficients of POEA-PAM/SDS system are somewhat less than those in their individual aqueous solutions at the same concentrations. This implies that the conformation of the copolymer in the solution has possibly undergone a change caused by the presence of SDS. It is worth noting that the phenoxy protons attached to the hydrophobic POEA moieties and S1, S3 protons of SDS show larger changes than the other protons of the mixed systems. At a concentration below the *cac*, we can observe that the T_2 values of the protons of the side-chain (typically of the phenoxy groups) have a relatively distinct change with the introduction of the SDS molecules. Their T_2 values decrease drastically with increasing SDS concentration. We observed that the T_2 values of the protons of SDS are long in the absence of POEA-PAM; however a large decrease can be observed on the introduction of POEA-PAM, which might be the result in the increase in viscosity of the solution. This implies that the variation in conformation of the hybrids solutions is due to the existence of SDS in the hybrids solutions. When the copolymer concentration is above the *cac*, 12 g L^{-1}, no

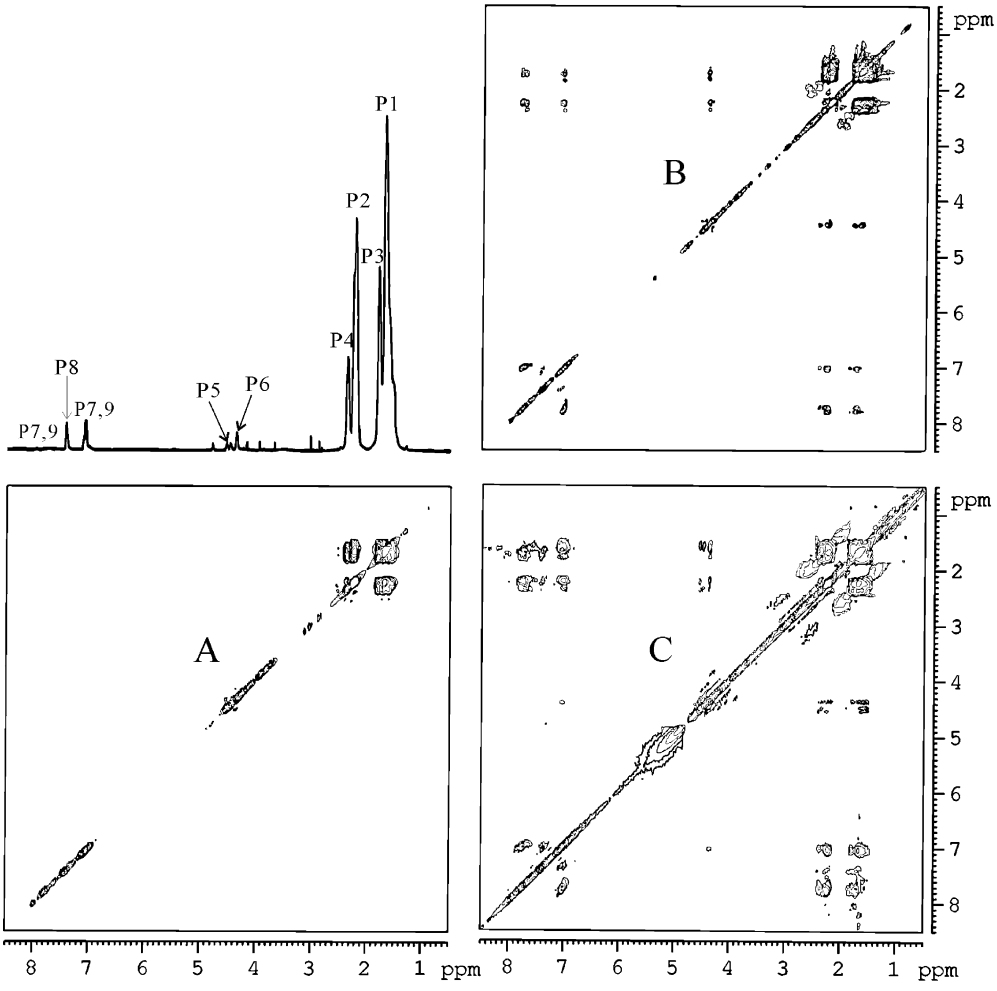

Fig. 4A–C Contour plots of the 2D nuclear Overhauser enhancement (NOESY) maps of POEA-PAM solution with a POEA content of 1.41 mol% at concentrations of **A** 3 g L^{-1}, **B** 7 g L^{-1}, and **C** 12 g L^{-1}, with a mixing times of 80 ms

Table 3 ^{1}H spin-spin relaxation time (T_2),T_R (T_2/T_1), and self-diffusion coefficients (D) with different SDS concentrations at a constant temperature of 298 K for the polymer 1 (POEA content is about 1.41 mol%). *Note*: Experimental error < 10%. *SDS* $NaSO_4CH_2CH_2(CH_2)_9CH_3$, (SDS protons numbered left to right::*S1*, S2, *S3*, *S4*)

[Polymer]	[SDS]	POEA-PAM					SDS						
		Ph		CH (P2,4)		$D\times10^{12}$	S1		S3		S4		$D\times10^{12}$
		T_2	T_R	T_2	T_R		T_2	T_R	T_2	T_R	T_2	T_R	
(g/L)	(mM)	(ms)		(ms)		m^2s^{-1}	(ms)		(ms)		(ms)		m^2s^{-1}
0	0.9	–	–	–	–	–	508	0.40	524	0.45	796	0.38	621
	9.0	–	–	–	–	–	336	0.30	372	0.38	665	0.37	392
	20	–	–	–	–	–	303	0.30	295	0.37	573	0.36	110
5	0	856	0.41	110	0.11	7.2	–	–	–	–	–	–	–
	0.9	738	0.36	108	0.11	6.2	382	0.32	417	0.38	607	0.30	450
	4.5	530	0.29	105	0.10	5.9	201	0.17	386	0.39	588	0.29	316
	9.0	692	0.37	90	0.09	8.4	320	0.27	308	0.32	500	0.30	352
	15	175	0.10	75	0.07	4.7	239	0.22	250	0.28	504	0.32	120
	20	134	0.08	69	0.07	2.4	216	0.22	219	0.24	511	0.30	80
12	0	576	0.31	97	0.09	3.5	–	–	–	–	–	–	–
	0.9	519	0.28	92	0.09	3.0	281	0.26	302	0.30	487	0.25	363
	4.5	482	0.27	87	0.09	2.8	225	0.23	278	0.26	474	0.25	298
	9.0	448	0.25	84	0.08	2.6	177	0.18	225	0.22	459	0.24	209
	20	501	0.27	89	0.09	1.7	182	0.18	249	0.25	471	0.25	110

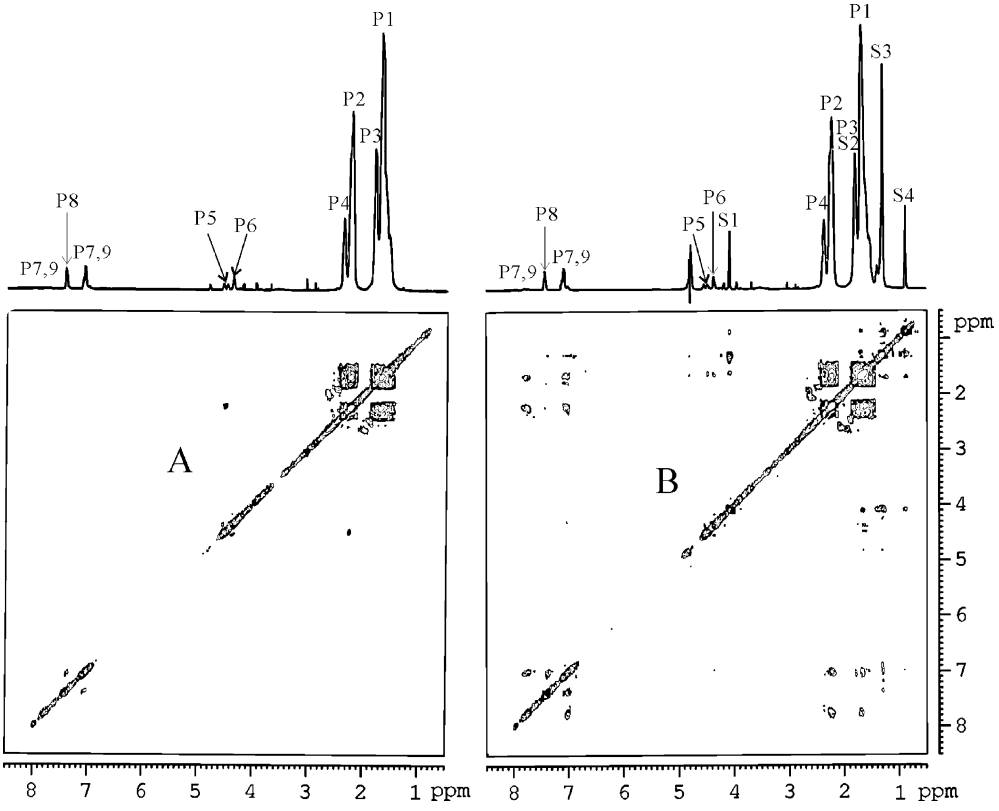

Fig. 5A, B Contour plots of the 2D NOESY maps of POEA-PAM solution with a POEA content of 1.41 mol% at a concentration of 5 g L^{-1} in the absence (**A**) and presence (**B**) of 0.9 mM sodium dodecyl sulfate (SDS)

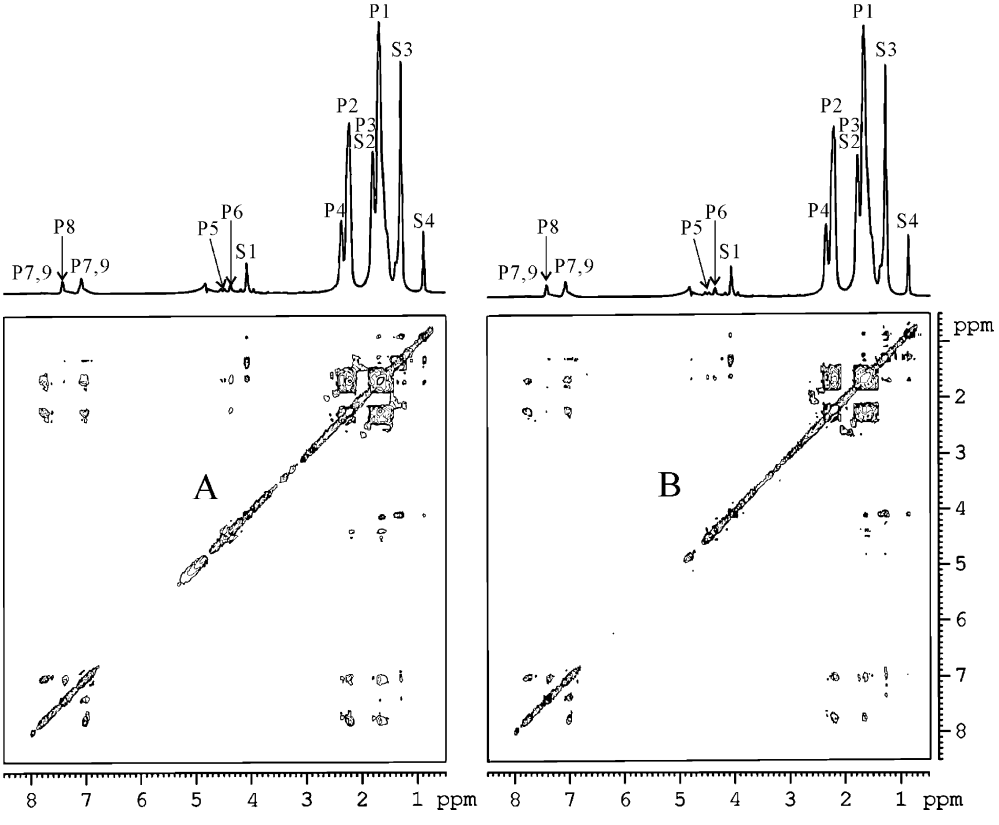

Fig. 6A, B Contour plots of the 2D NOESY maps of POEA-PAM solution with a POEA content of 1.41 mol% at a concentration of 12 g L^{-1} at SDS concentrations 0.9 mM (**A**) and 4.5 mM (**B**)

similar variation was observed. This suggests that the POEA-PAM aggregate is stable, and almost unaffected by the introduction of SDS. The self-diffusion coefficient measurements are in good agreement with the relaxation results.

The interaction between the two solutes can also be demonstrated by the 2D NOESY measurements. The results are shown in Fig. 5 and Fig. 6. Figure 5 shows the 2D NOESY contour plots of the polymer 1 at a concentration below its *cac* (5 g L^{-1}) in the absence and presence of 0.9 mM SDS with a mixing time of 80 ms. It is obvious that more cross-peaks between the protons of the copolymer appear in the presence of SDS than do in the absence of SDS, i.e., cross-peaks among phenoxy protons, and between the main chain protons and phenoxy protons appear in the 2D NOESY map in the presence of SDS. This indicates that the introduction of SDS decreases the *cac* of the copolymer and reinforces the aggregation formation ahead. Furthermore, the appearance of cross-peaks between the POEA-PAM protons and SDS protons (P1-S1, P1-S4, and phenoxy protons and S3) is direct evidence of the interaction between POEA-PAM and SDS. Any difference between the 2D NOESY maps (Fig. 6A, B) of polymer 1 at a concentration above its *cac* (12 g L^{-1}) with different SDS concentrations (Fig. 6) is barely visible. This agrees well with the results of relaxation measurements (Table 3).

To conclude, the results obtained in the present study clearly show that the hydrophobic POEA moieties of the novel hydrophobically associating copolymers POEA-PAM associate in a definite narrow concentration range between the phenoxy rings and the backbones. In the POEA-PAM aggregates, the hydrophobic phenoxy moieties are possibly located in the vicinity of the PAM backbones. The content of hydrophobic POEA moieties affects the dynamics of copolymer aggregations, i.e., the lower the hydrophobic POEA content, the more the motion is restricted and the more stable are the aggregates. The anionic surfactant SDS shows a significant interaction with the copolymer in the dilute solution, which decreases the *cac* of the copolymer and reinforces the aggregation of the POEA-PAM monomer. In the SDS/POEA-PAM mixed systems, the hydrophobic methylene groups of the surfactant SDS molecules are preferentially located near the phenoxy groups of the POEA hydrophobes. The self-diffusion coefficients, relaxation measurements, and 2D NOESY experiments show that the POEA-PAM aggregates in concentrated solutions are little affected by the introduction of SDS.

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