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ORIGINAL CONTRIBUTION

Rheological behavior of PAA–C_nTAB complex: influence of PAA charge density and surfactant tail length in PAA semidilute aqueous solution

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Abstract Interactions between anionic polyelectrolyte, poly(acrylic acid) (PAA), and cationic surfactant, alkyltrimethylammonium bromide (C_nTAB), were investigated by rheological measurements in semidilute PAA solution. The dependences of the rheological behavior on the chain length of the surfactant, PAA neutralization degree, and temperature were discussed. The results revealed that both dodecyl and cetyltrimethylammonium bromides (C_{12}TAB and C_{16}TAB) could increase the viscosity of PAA solution when the surfactant amounts surpassed a critical surfactant concentration (C_c), and C_c of C_{16}TAB was lower than that of C_{12}TAB at same PAA neutralization degree. The increase of viscosity is attributed to the surfactant micelles bridging of the polymer chains and confine the mobility PAA chain. On the other hand, it is found that the hydrogen bonding also played an important role in the PAA–C_nTAB system, especially in lower neutralization degree PAA solution, which results in the viscosity increase rapidly with the added surfactant into lower neutralization degree PAA solution.

Keywords Polyelectrolyte · Surfactant · Micelle · Viscosity · Hydrogen bonding

Introduction

Polymer and surfactant are widely used in industrial situation such as coating fluids, laundry products, pharmaceuticals, and cosmetics due to their excellent individual and cooperative functions [1]. Surfactants are generally used for emulsifying and solubilizing the immiscible substances, while polymers are often employed to control the rheological properties of solutions and suspensions. However, when polymers and surfactants are used together, they would interact with each other and therefore lead to some unforeseen effects, namely, the interaction could bring both good and bad results. For example, the addition of surfactant to aqueous polymer solution would not only remarkably decrease critical micelle concentration (CMC) of the surfactant [2–4], but also change to different extents of rheological properties for the aqueous polymer solution [5, 6]. The polymer–surfactant complex would be formed and sometimes precipitate from the mixtures and then making no performance [7, 8]. Hence, the interaction between surfactants and polymers in aqueous colloidal systems is very important. In particular, the interaction between polyelectrolyte and surfactant with opposite charges [2, 9–12], for its application in electrostatic self-assembly, has attracted more and more researchers' attention [1, 13–15].

The anionic polyelectrolyte poly(acrylic acid) (PAA) and two cationic surfactants dodecyl and cetyltrimethylammonium bromides (C_{12}TAB, and C_{16}TAB) were chosen in this work, since PAA is a well-defined weak polyacid and its charge density can be modified by controlling neutralization. Moreover, C_{12}TAB and C_{16}TAB are popular surfactants and their individual properties are well known. Furthermore, the research results concerning the PAA–C_nTAB system in PAA

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dilute solution [5, 16, 17] can be used to compare with our results.

Except for the electrostatic interactions, the hydrogen bonding and hydrophobic interactions between PAA and C_nTAB have been also believed to play an important role in PAA–C_nTAB complex formation. Kiefer et al. [18] reported that the onset of binding tetradecyltrimethylammonium bromide (TTAB) with PAA or poly(methacrylic acid) (PMA) was significantly influenced by the changes of polyelectrolyte conformation and hydrophobicity. Yoshida et al. [19, 20] studied the effect of pH on electrostatic interaction and hydrogen bonding between PAA and cetyltrimethylammonium chloride (CATC)/octaethylene glycol dodecyl ethers ($C_{12}E_8$) mixed micelles and found that the hydrogen bonding dominated the interactions in the case of low pH. Wang et al. [21] reported that there was a critical neutralization degree (α_c) in surfactant added polyelectrolyte solution system, in which α is the neutralization degree of PAA, defined as [NaOH]/[PAA], the PAA mole concentration is expressed as monomers. When $\alpha < \alpha_c$, PAA–C_{12}TAB binding was by the hydrogen bonding, while $\alpha > \alpha_c$, the hydrogen bonding become less dominant as PAA was progressively ionized and C_{12}TAB binds to the charged polymer chains driven by electrostatic attraction.

Addition of the opposite-charged surfactants to the polyelectrolyte dilute solution could make the viscosity decrease [5, 17, 22, 23], which is ascribed to the drop of the effective charge density of polyelectrolyte and the increase of the solution ionic strength. Furthermore, Lim et al. [17] found C_{16}TAB would make dilute aqueous PAA (M_η= 750 kDa) solution exhibit nearly Newtonian behavior from shear thinning behavior in pure PAA solutions. On the basis of the scaling theory [24, 25], Colby et al. [5] established a simple model for describing the viscosity change of opposite charge polyelectrolyte-surfactant binding in dilute and unentangled semidilute polyelectrolyte solutions in good solvent. A quantitative prediction of solution viscosity could be carried out for strongly charged polyelectrolyte with no adjustable parameters using Colby's model. Our previous work [26] on sodium carboxymethyl cellulose (NaCMC)-C_{16}TAB system demonstrated that the viscosity change of unentangled semidilute NaCMC solution was dependent on the critical aggregation concentration (CAC) and saturated concentration (C_{sc}) of C_{16}TAB.

It is well known that the surface tension [23, 27], conductivity [28, 29], microcalorimetry [21, 30, 31], fluorescence [14, 29, 32], light scattering [16, 33], and NMR [34] have been extensively used to detect the interactions between polyelectrolyte and surfactant in dilute aqueous polyelectrolyte solution. However, as far as we know, few works concerning in polyelectrolyte-surfactant interactions in semidilute polymer solutions have been reported. An important reason is the above measurements would disable due to the poor light transmission, high viscosity, and high conductivity. It is noted that the rheological measurement is believed to be a preferred approach for examining the interactions between polymer blends [35–37]. However, to our knowledge, rheological measurement has been seldom employed to study surfactant–polymer systems of opposite charges in semidilute polyelectrolyte solutions.

The purpose of the present article is to probe the rheological behavior of opposite charge polyelectrolyte-surfactant systems under shear in semidilute polyelectrolyte solution. The rheological behavior was measured as function of surfactant tail length, PAA charge density, and temperatures.

Experiments

Materials

Anionic polyelectrolyte PAA (stocked in form of aqueous solution with 35 wt.%, $M_w = 3\times10^4$ g/mol) was acquired from Aldrich. Cationic surfactants C_{12}TAB and C_{16}TAB were also purchased from Aldrich, their CMC measured by conductivity are 14 and 1 mM at 25 °C, respectively. All solutions were prepared using deionized water and all materials were used without further purification.

Sample preparation

First, PAA were prepared into stock solutions with deionized water and then titrated with NaOH to the desired degree of neutralization (α=0, 0.2, 0.4, 0.6, 0.8, and 1.0). PAA solutions with different concentrations were diluted from the PAA stock solutions. PAA–C_nTAB system solutions were prepared by mixing appropriate aqueous solutions of polyelectrolyte and surfactant at certain ratio to gain the desired composition. The surfactant solution with certain concentration was slowly added into PAA solution using a micropipette while the solution was gently stirred, in order to avoid the premature precipitation of surfactant at locally high surfactant concentration. The final solutions with certain concentrations were obtained by adding deionized water. To gain complete dissolution and equilibrium, the prepared solutions were hold 48 h at room temperature before test. It is noted that PAA solution with neutralization degrees of 0.2 and 0.4 would precipitate when some amount of C_nTAB stock solution were added. PAA–C_nTAB system with PAA neutralization degree 0.6, 0.8, and 1.0 were used here.

Measurements

The rheological tests were performed by using an AR-G2 rheometer (TA instrument, USA) with geometry of cone

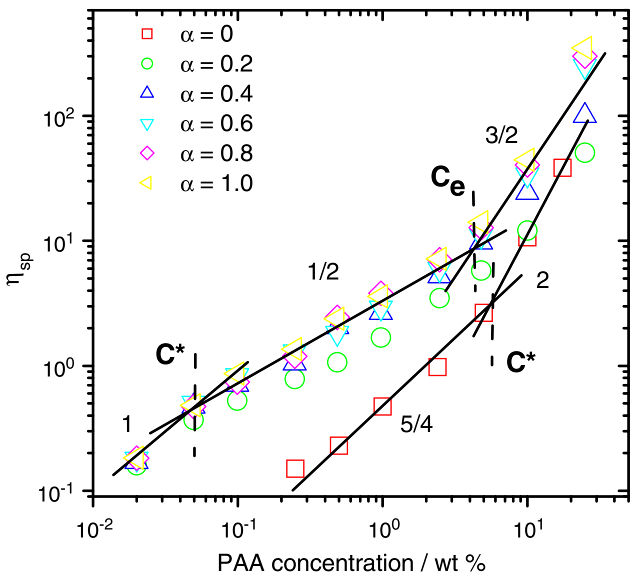

Fig. 1 Dependence of specific viscosity (η_{sp}) on concentrations of PAA solutions with different neutralizations

and plate (40 mm). As a stress-controlled rheometer, AR-G2 has a minimum torque of 0.01 μN m for low-viscosity measurements. The influence of shear rate was tested by a shear flow mode and the shear rates were from 1 to 1,000 s^{-1}. The viscosity dependence on temperature was tested form 10 to 45 °C at a shear rate of 100 s^{-1}. Because all the samples exhibited Newtonian behavior in the shear rate from 1 to 1,000 s^{-1}, viscosity at the shear rate of 100 s^{-1} could be represented as the zero shear rate viscosity (η_0). The temperature tests were repeated twice for one sample, and the curve of viscosity–temperature was completely superposed, indicating the fact that there was no volatilization of solvent in the studied temperature region.

Tensiometric measurements were taken on a calibrated tensiometer (AF-02, Aofang Instrument, China) using the maximum bubble pressure method. 100 ml PAA solution with concentration 10 wt.% (1.38 M, mole concentration of PAA monomer) was put into a thermostatic container, and then 20 mM $C_{12}TAB$ or $C_{16}TAB$ solution was added stepwise to the PAA solution with a micropipette, gently stirring. An interval of 5 min was adopted for equilibration during measurements.

Results and discussion

Rheological behavior of PAA solution

Figure 1 presents the specific viscosity (η_{sp}) of aqueous PAA solutions with various neutralization degrees at different concentrations. The PAA solutions can be grouped into two categories: PAA (α=0, neutral polymer) and PAA (α=0.2, 0.4, 0.6, 0.8, and 1.0). For aqueous PAA solution with α=0, the dependence of η_{sp} on PAA concentration (C_p) can be divided into two parts by an overlap concentration (C^*): dilute and semidilute solutions. On the other hand, for all the neutralized PAA solutions, η_{sp} increases with increasing of C_p and can be divided into three parts depending on C^* and entangled concentrations (C_e). When C_p < 0.05 wt.% and C_p > 4.2 wt.%, η_{sp} remarkably increases with C_p, while in the medium C_p region (0.05 < C_p < 4.2 wt.%), η_{sp} increases slightly with C_p. According to Dobrynin's scaling theory [24, 25, 38] and the rheological behavior of polyelectrolyte solution reported by Colby [39, 40], $\eta_{sp} \sim C_p$ holds in dilute regime ($C_p < C^*$), $\eta_{sp} \sim C_p^{1/2}$ in the semidilute unentangled regime ($C^* < C_p < C_e$), and $\eta_{sp} \sim C_p^{3/2}$ in semidilute entangled regime. It can be seen that the experimental data follow basically the scaling plots, thus, the two intercept points correspond to C^* and C_e could be obtained. To examine the rheological behavior of PAA and surfactant with opposite charge in entangled semidilute PAA solution, PAA solution with concentration of 10 wt.% (1.38 M) is chosen for next tests.

CAC of C_nTAB added to entangled semidilute PAA solution

In the presence of polymer, micelle-like surfactant aggregates start to form along the polymer chain at CAC in solution. Thus, CAC is the threshold of concentration for surfactant binding to polymer chain. A characteristic of

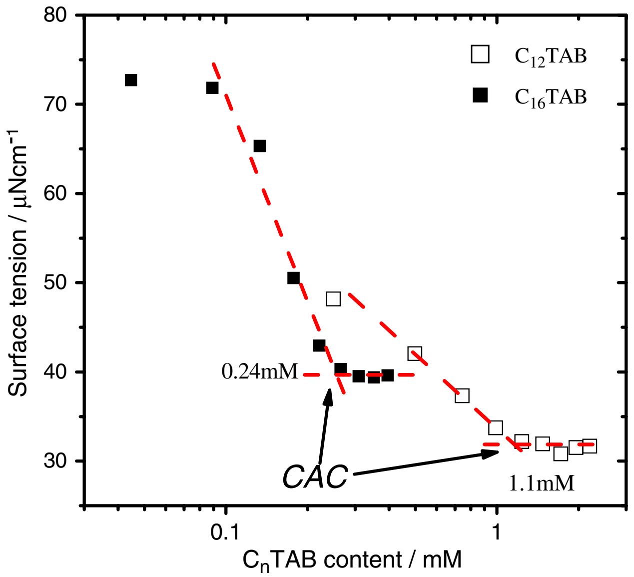

Fig. 2 Dependence of surface tension on $C_{12}TAB$ and $C_{16}TAB$ concentration for 10 wt.% PAA aqueous solution with neutralized degree of 0.6

Table 1 Parameters of CAC, C_c, C_c-CAC *and* (C_c-CAC)/*N* for C_{12}TAB and C_{16}TAB added into 10 wt% PAA solutions with different neutralization degrees, respectively

	α	CAC (mM)	C_c (mM)	C_c–CAC (mM)	(C_c–CAC)/*N* (mM)
C_{12}TAB	0.6	1.10	9.7	8.60	0.145
	0.8	1.28	9.6	8.32	0.141
	1.0	1.15	9.8	8.65	0.147
C_{16}TAB	0.6	0.24	4.0	3.76	0.035
	0.8	0.24	4.8	4.56	0.043
	1.0	0.29	4.2	3.91	0.037

CAC is that it is much lower than the CMC of the corresponding surfactant [2, 4, 11, 29, 41]. The CAC is an important parameter to evaluate the interaction of surfactant and polymer. Figure 2 gives the dependence of surface tension on C_{12}TAB and C_{16}TAB concentration (C_s) for 10 wt.% aqueous PAA solution with neutralized degrees of 0.6. The beginning concentration of the plateau corresponds to the CAC [1]. The CAC of C_{12}TAB and C_{16}TAB in 10 wt.% PAA with α=0.6, at 25 °C, are 1.1 mM and 0.24 mM, respectively. CAC of C_{12}TAB and C_{16}TAB add in 10 wt.% PAA with α=0.6, 0.8 and 1.0 are listed in Table 1. It is found that the CAC of C_{16}TAB is lower than that of C_{12}TAB and CAC is independent of PAA neutralization degree.

Rheological behavior of PAA–C_nTAB system

Influence of surfactant tail length

Figure 3 presents the shear rate dependence of apparent viscosity (η_a) at 25 °C for PAA solutions (10 wt.%, α=0.8) at different C_{16}TAB concentrations. The viscosity of PAA solutions do not perform shear thinning behavior in the shear rate region tested, which results from the low molecular weight of PAA. The addition of C_{16}TAB does not affect the shear rate dependence of rheological behavior of PAA solution, and PAA–C_{16}TAB systems still exhibit Newtonian behavior in the studied shear rate region. Other PAA–C_nTAB systems also exhibit Newtonian behavior. Hence, the viscosities discussed as follows are all considered as η_0.

Figure 4 shows the influence of C_{12}TAB and C_{16}TAB concentration on η_0 of PAA solutions with different neutralization degrees. It is obvious that the dependence of η_0 on surfactant concentration (C_s) is divided into two different regions. At lower C_s, η_0 changes slightly with C_s increasing. When C_s reaches a critical concentration (C_c), η_0 increases remarkably. The increase of η_0 is attributed to the surfactant micelles bridging of the polymer chains and confine the mobility of PAA chain. The phenomenon is also found in some previous reports [26, 42]. It should be noted that the C_c of C_{16}TAB added to PAA solution is lower than C_c of C_{12}TAB. Moreover, for the same neutralization degree PAA solution, the slopes of logη_0 versus logC_s plots for both C_{12}TAB and C_{16}TAB are nearly the same in $C_s > C_c$ region. This suggests that the scaling relationship between η_0 of PAA–C_nTAB system and C_s is independent of surfactant tail length. C_c of C_{12}TAB and C_{16}TAB added in 10 wt.% PAA with α=0.6, 0.8 and 1.0 are listed in Table 1. It is found that the C_c of C_{16}TAB or C_{12}TAB is independent of PAA neutralization degree.

The maximum added C_nTAB concentration in PAA solutions is only 50 mM in this work, whereas the acrylic acid monomer concentration of PAA is $\alpha\cdot1.38$ M (at least $1.38\times0.6=0.83$ M). This means that the neutralized acrylic acid monomers are always excessive in all PAA–C_nTAB system solutions. So, when C_s > CAC, the increasing C_nTAB will just form micelles bound to PAA chains, and no free micelles exist in the system. From Table 1, one could find that C_c > CAC, indicating that the viscosity increase of PAA solution does not start at CAC, and needs a certain amount of C_nTAB micelles. Because CAC is the beginning concentration for surfactant binding to polymer chain and forming micelles, the number of C_nTAB

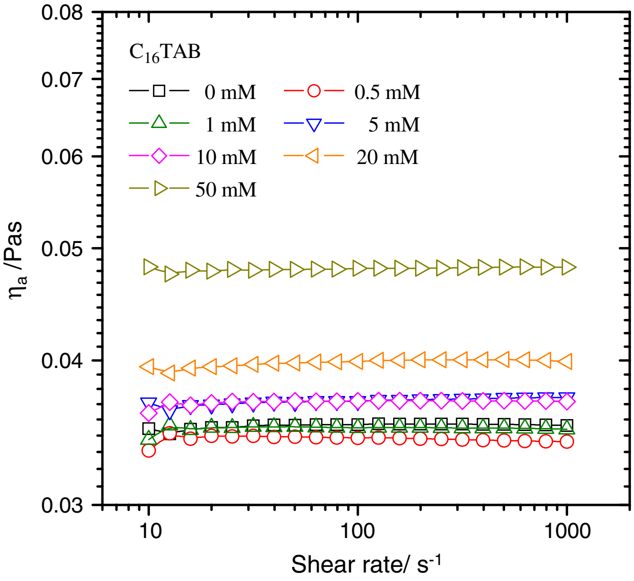

Fig. 3 Dependence of apparent viscosity (η_a) on the shear rate at 25 °C for 10 wt.% PAA solutions (α=0.8) with different C_{16}TAB concentrations added

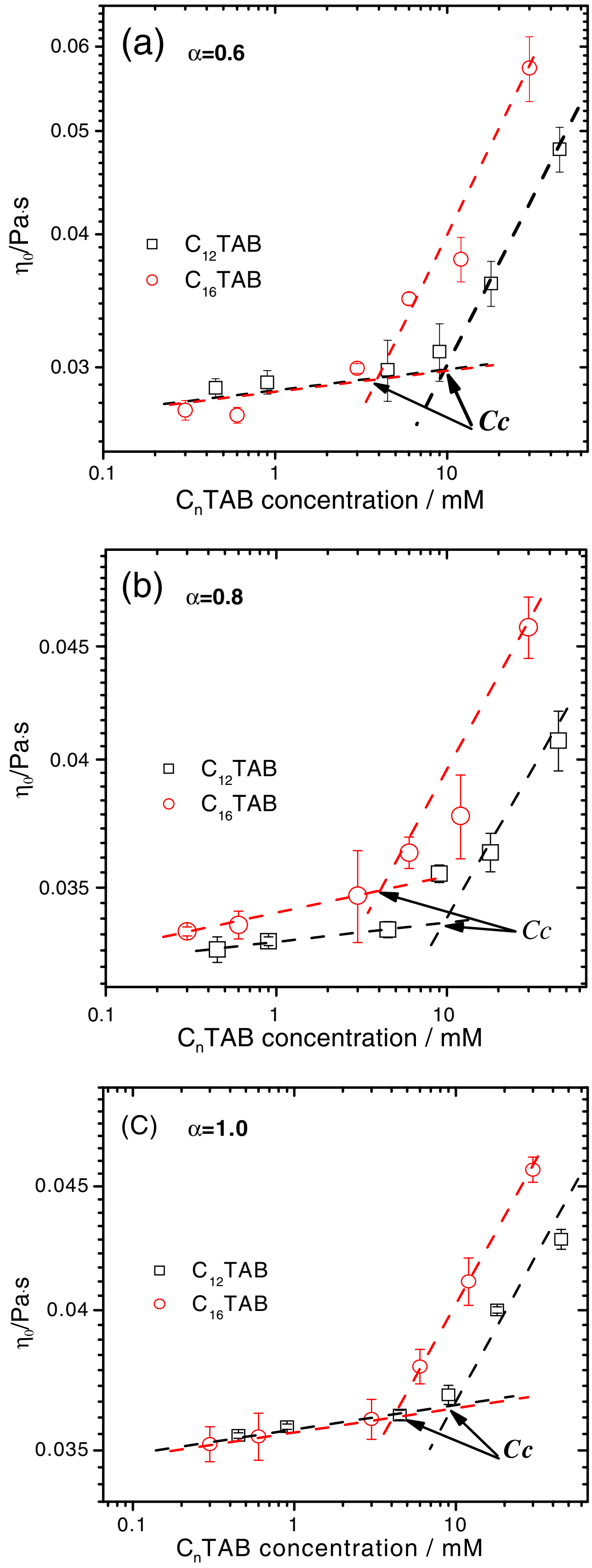

Fig. 4 Dependence of zero shear viscosity (η_0) on surfactant (C_{12}TAB and C_{16}TAB) concentrations for 10 wt.% PAA solution with different neutralization degree of **a** 0.6, **b** 0.8, and (**c**)1.0

monomers in micelles bound to PAA chains can be estimated as C_c–CAC at C_c. C_c–CAC may be the threshold C_s to form physical networks by bridging PAA long chains. In addition, the aggregation number (N) for C_{12}TAB and C_{16}TAB micelle is 58 [41] and 107 [16], respectively, which is considered to be equivalent in aqueous solution either with or without polyelectrolyte [41]. Therefore, (C_c–CAC)/N, the beginning micelle concentration in order to

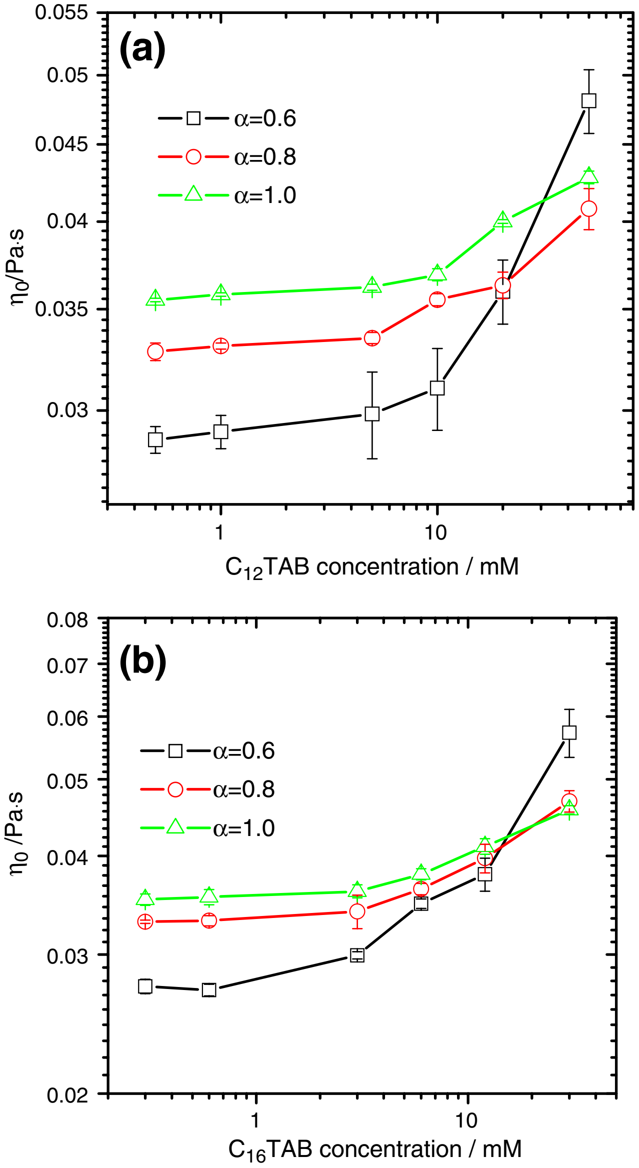

Fig. 5 Dependence of zero shear viscosity (η_0) on surfactant concentration for 10 wt.% PAA solution with different neutralization degrees **a** C_{12}TAB and **b** C_{16}TAB

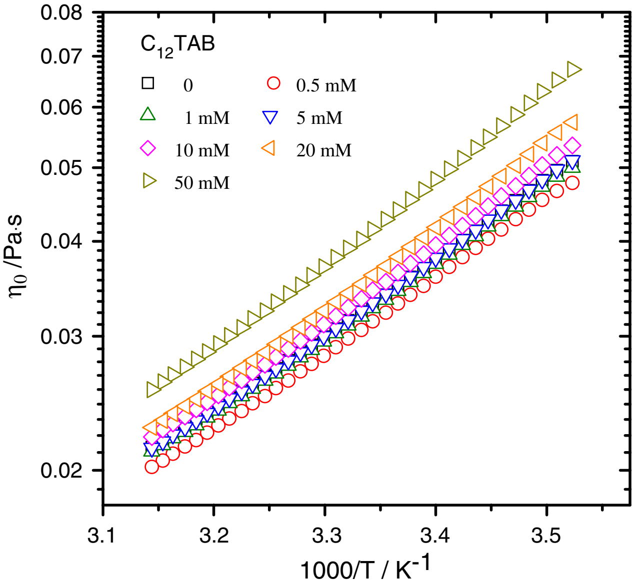

Fig. 6 Dependence of zero shear viscosity (η_0) on temperature reciprocal for 10 wt.% PAA (α=0.8) aqueous solution with different concentration of C$_{12}$TAB in a log-normal plot

form physical networks by bridging PAA long chains can be calculated. Table 1 presents C_c–CAC and $(C_c$–CAC$)/N$ for C$_{12}$TAB and C$_{16}$TAB added to PAA solutions with different neutralization degrees. It is found that both C_c–CAC and $(C_c$–CAC$)/N$ of C$_{16}$TAB are lower than those of C$_{12}$TAB. It is well known that the size of C$_{16}$TAB micelle size is larger than that of C$_{12}$TAB. Table 1 indicates that the micelle size plays an important role in forming physical networks of the system, as the micelle size increases, threshold C_s to form physical networks decreased.

Influence of PAA charge density

Figure 5 gives the dependence of viscosity on surfactant concentration for PAA solution with different neutralized degrees. It can be seen that η_0 of the PAA–C$_n$TAB systems with different neutralization degree increases slightly when $C_s < C_c$ and remarkably when $C_s > C_c$. Moreover, for α= 0.6, η_0 of the PAA–C$_n$TAB system increases much faster in $C_s > C_c$ region than that for other higher neutralization degrees, such as α=0.8 and 1.0. When C_s > 30 mM and α= 0.6, η_0 of PAA–C$_n$TAB system is even higher than that of PAA solutions with α=0.8 or 1.0. These results indicate that when $C_s > C_c$, η_0 of PAA–C$_n$TAB system with lower neutralization degree is much more sensitive to C_s than that with higher neutralization degree. The increase of η_0 is attributed to the surfactant micelles bridging of the polymer chains and confine the mobility PAA chain. It is worth noting that PAA–C$_n$TAB interactions include electrostatic interaction, hydrogen bonding, and hydrophobic interaction. Electrostatic interaction occurs between the opposite charge of PAA and C$_n$TAB, and hydrogen bonding exists between un-neutralized carboxyl of PAA chains and surfactant, while the hydrophobic interaction results from the attraction between PAA backbone and the surfactant alkyl chain. In the present work, since PAA is always excessive, the electrostatic interaction is actually decided by the amount of C$_n$TAB. Hence, all the complexes should have the same electrostatic interactions if C_s is fixed. The lower neutralization degree PAA solution means lower neutralized carboxyl acid fraction, i.e., higher carboxyl acid group fraction in PAA chains. It is well accepted that the

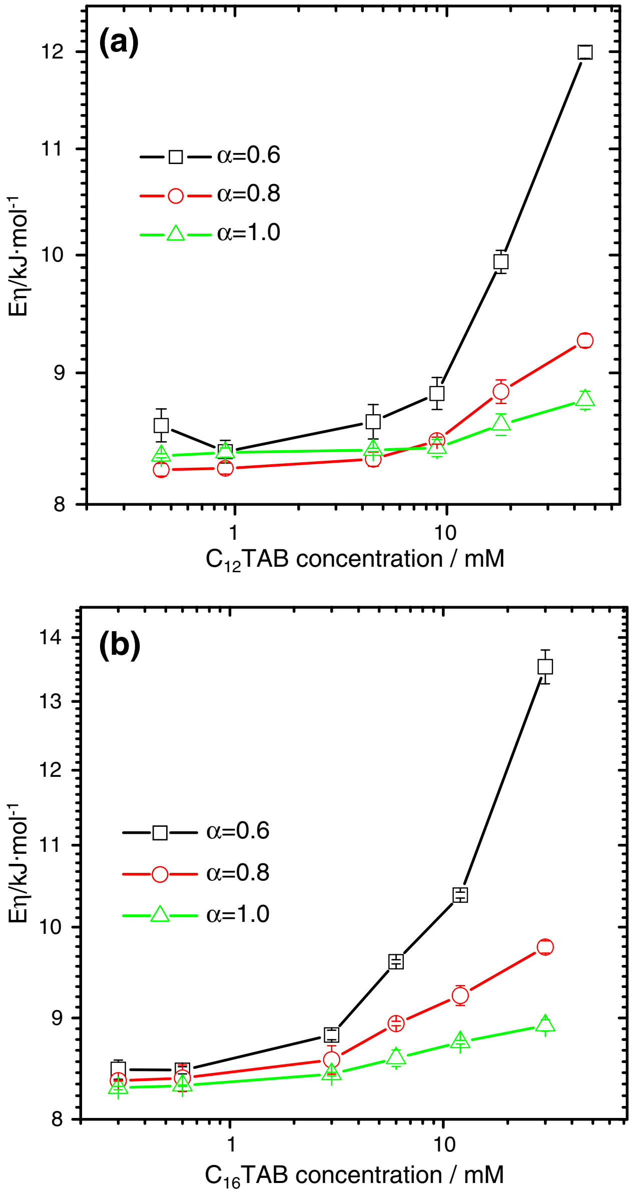

Fig. 7 Dependence of viscous flow activation energy (E_η) on surfactant concentration **a** C$_{12}$TAB and **b** C$_{16}$TAB for 10 wt.% PAA solution with different neutralization degrees of 0.6, 0.8, and 1.0

hydrogen atom in carboxyl can form hydrogen bonding with electronegative atom [43]. The C$_n$TAB molecule has nitrogen and bromine (C$_n$TAB micelles are partly neutralized) atoms, therefore, PAA chains can form hydrogen bonding with C$_n$TAB micelles. In addition, because the alkyl chains of surfactant bound to PAA chains are almost in the interior of the micelles, the hydrophobic interaction can be neglected in the present systems. Therefore, the hydrogen bonding plays an important role in PAA–C$_n$TAB system. Since the hydrogen bonding content would increase with the decrease of PAA neutralization degree, it is reasonable that the η_0 of lower neutralization degree PAA solution increases much faster with C_s is attributed to the higher fraction of hydrogen bonding in the system.

Viscous flow activation energy (E_η) of PAA–C$_n$TAB system

It is generally accepted that the hydrogen bonding is temperature-sensitive [43], hence, the viscosity test with increasing temperature can be used to verify whether there are more hydrogen bondings in lower neutralization degree PAA–C$_n$TAB systems. Figure 6 gives the temperature dependence of the viscosity of aqueous PAA solution (α= 0.8) with different C$_{12}$TAB concentrations. It can be seen that all plots of logη_0 versus $1/T$ exhibit linear characteristics. η_0 of other PAA–C$_n$TAB systems also show the linear characteristics. Therefore, the flow activation energy (E_η) can be calculated by using the Arrhenius equation $\eta = A\mathrm{e}^{-\mathrm{E}_\eta/\mathrm{RT}}$. E_η is always used to evaluate the temperature sensitivity of viscosity for polymeric liquid.

Figure 7 presents the dependence of E_η on C_s in different neutralized PAA solutions. It can be seen that E_η increases with both the increase of C_s and the decrease of PAA neutralization degree. This indicates that PAA–C$_n$TAB system is more temperature-sensitive at low-neutralization degree of PAA solution and high C$_n$TAB concentration. This result confirms that the lower neutralization degree PAA–C$_n$TAB solutions have more hydrogen bondings and suggests that the hydrogen bondings that increase the PAA viscosity at low-neutralization degree are rational.

Conclusion

Interactions between PAA and C$_n$TAB were investigated by rheological measurements in semidilute PAA solution. Different from added opposite-charged surfactants to polyelectrolyte dilute solution making the viscosity decrease, both C$_{12}$TAB and C$_{16}$TAB increase the viscosity of PAA semidilute solution when the surfactant amounts surpassed C_c. The results present that the viscosity increasing of PAA solution does not start at CAC and needs a certain amount of C$_n$TAB micelles. The increase of PAA solution viscosity is attributed to the surfactant micelles' bridging of the polymer chains and confine the mobility PAA chains. On the other hand, the hydrogen bonding played an important role in the PAA–C$_n$TAB system, especially in lower neutralization degree PAA solution. The temperature test verified that more hydrogen bonding formed in lower neutralization degree PAA solution with C$_n$TAB.

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