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Complex formation between hydroxypropylcellulose and hexadecyltrimethylamonium bromide as studied by light scattering and viscometry

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Abstract

Complex formation between a semiflexible nonionic polymer, hydroxypropylcellulose (HPC), and a cationic surfactant, hexadecyltrimethylamonium bromide (HTAB), is investigated by static and dynamic light scattering (DLS) and by viscometry. Upon addition of surfactant, at a fixed polymer concentration, the solution specific viscosity increases initially to a maximum value, and then decreases, in parallel with the hydrodynamic radius of the polymer–surfactant complex measured by DLS. The increase in specific viscosity and hydrodynamic radius is interpreted to reflect chain expansion due to the electrostatic interaction between bound micelles. The maximum occurs at the saturation of micelle binding, and the subsequent decrease, as more surfactant is added, is due to chain contraction because of electrostatic screening by free micelles and their Br^- counter-ions. This behavior was observed at various polymer concentrations and we found that the maximum in the solution viscosity and in the hydrodynamic radius occurs always at [HTAB]/[HPC] = 0.18, from which we deduce that the average number of surfactant molecules bound to a single HPC chain is 28. The effect of electrostatic interactions between the bound micelles on the solution viscosity can be represented by the "interaction viscosity", η_I, defined as the difference between the measured viscosity of the ternary polymer solution and that computed from the sum of solvent, polymer and surfactant. We find that the normalized interaction viscosity $\eta_I/\eta_{I,max}$, where $\eta_{I,max}$ is the interaction viscosity at the maximum surfactant–polymer binding, is a common function of [HTAB]/[HPC], independent of polymer concentration and ionic strength. The origin of this observation appears to lie in the fact that the fraction of complex formed and the relative chain expansion are functions only of the ratio [HTAB]/[HPC].

Keywords: Hydroxypropylcellulose; Hexadecyltrimethylamonium bromide; Surfactant–polymer binding

1. Background

Polymer–surfactant interactions are important in many industrial applications, such as paints and coating, detergent processing, cosmetic product formulation, and in ternary oil recovery. Recent reviews of polymer–surfactant solutions and interactions may be found in several published articles [1–3]. The main driving force for polymer–surfactant interaction is believed to be reduction of the interfacial area between the hydrophobic polymer segments and the aqueous solvent by association of these segments with the exposed hydrophobic parts of aggregating surfactants. Here the hydrophobicity of the polymer chain plays a major role, although the perturbation of the hydration sheath by the surfactant head groups and counter-ions may influence the free energy transfer of the polymer from the aqueous to the micellar phase. Since the hydrophobic polymer strands are believed to replace water molecules structured around the micellar headgroups, stabilization of the micelle upon binding to polymer results from a reduction of interfacial tension between the hydrophobic core and water. Further, specific interactions such as hydrogen bonding between the polymer and headgroups may also be a factor [4].

In a system of fixed polymer concentration with increasing amount of surfactant, no interaction between the polymer and surfactant is detected until a critical aggregation concentration (CAC) is reached. Therefore, the CAC is a surfactant concentration at which an interaction between polymer and surfactant takes place and a complex starts to form. Generally, it appears that an ionic surfactant interacts with the polymer chain at a CAC, which is substantially lower than the critical micelle concentration (CMC) of the pure surfactant solution [5].

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Various *structures* have been proposed for the complexes formed between a nonionic polymer and an ionic surfactant [6]: (a) redistribution of the surfactant between the bulk solution and the coil regions; (b) surfactant molecules bound individually along polymer chain; (c) surfactant molecules cluster around hydrophobic sites on the polymer chain; and (d) polymer molecules wrap around surfactant micelles in such a way that the polymer segments partially penetrate and wrap around the polar headgroup regions of the micelles. Experimental small-angle neutron scattering (SANS) studies suggest a "necklace and bead" structure for polyethylene oxide–sodium dodecyl sulfate (PEO–SDS) complexes [7–9]. These results are consistent with the models (c) and (d) above.

The interaction between nonionic water-soluble polymer and cationic surfactants has been observed so far to be nonexistent or very weak, contrary to the case of anionic surfactants where many investigations have shown clear evidence of interaction [10]. This behavior has been explained as due to: (a) the bulkiness of the cationic headgroup [11]; (b) the electrostatic repulsion between polymer and surfactant due to the possible positive charge of polymer upon protonation [12]; and (c) the hydration shell of the polymer which favors interaction with anionic surfactant [13]. However, there is an evidence of complex formation between hydroxypropylcellulose (HPC) and hexadecyltrimethyamonium chloride (HTAC) micelles, on the basis of fluorescence probe and label experiments [14]. Likewise, there was an interaction between ethyl (hydroxyethyl) cellulose (EHEC) and two cationic surfactants, hexadecyltrimethylamonium chloride and bromide (HTAC and HTAB), as demonstrated by means of electrical conductivity and chloride ion self-diffusion measurement for HTAC and by time-resolved fluorescence quenching for HTAC and HTAB [15]. The complex formation between nonionic polymer PPO and cetyltrimethylamonium surfactant molecules (CTAX, $X^- = Cl^-$, ClO_3^-, NO_3^-) were investigated by means of several techniques [4]. The presence of a polymer gives rise to a CAC lower than CMC, a higher micellar ionization degree, and a smaller N for the bound micelles in the presence of PPO. The influence of polymers on the micellization of cetyltrimethylamonium salts was investigated [16]. The critical micelle concentration values and aggregation numbers of the cetyltrimethylamonium bromide (CTAB) micelles were measured in the presence and absence of poly(vinyl methyl ether) (PVME), and poly(vinyl pyrrolidone) (PVP). Association of the micelles with PVME and PVP is apparent from a reduction in both the CMC and the aggregation number. From viscosity measurements in the presence of PVME, it was deduced that rod-like micelles of hexadecyltrimethylamonium salicylate (HTASal) and tosylate (HTATS) transform to spherical polymer-bound micelle. Other formations of complexes between nonionic polymers and cationic surfactants have also been reported [17,18].

Hydroxyproplycellulose is water-soluble at room temperature. At elevated temperatures a lower critical solution temperature (LCST) is observed [14,19]. CTAB or HTAB ($C_{13}H_{33}N(CH_3)_3Br$) is a cationic surfactant. We present below evidence for complex formation between HPC and HTAB, through dynamic light scattering (DLS) and viscosity measurements. As the amount of surfactant in HPC solutions is increased, a maximum is observed in both the specific viscosity and the DLS hydrodynamic radius. The results are interpreted as indicative that chain expansion followed by contraction occurs through a polyelectrolyte effect on formation of a polymer–surfactant complex.

2. Experimental section

2.1. Materials

Hydroxypropylcellulose (HPC) of nominal molecular weight 100 000 was purchased from Aldrich Chemical Co. and used without further purification. Hexacetyltrimethylamonium bromide (HTAB) was purchased from Aldrich Chemical Co. The molecular weight of HTAB is 346. Sterile water, purchased from Government Pharmaceutical Organization, was filtered three times with 0.22 μm Millipore filters prior to use. Analytical grade sodium chloride (NaCl), purchased from P & N Company, was used to vary the solution ionic strength.

2.2. Preparations

Polymer stock solution was prepared at room temperature by dissolving HPC in powder form in sterile water and by gentle stirring for a period of 3–5 days. The stock solution was filtered once with a 0.45 μm Millipore filter and stored at 10°C. Surfactant and polymer–surfactant solutions were prepared by adding appropriate amounts of HTAB into mixtures of sterile water and polymer stock solutions and by gentle stirring for at least 24 h. Prior to viscometric and light scattering measurements, all sample solutions were centrifuged at 30°C at the speed of 10 000 rpm for 60 min and then filtered with 0.22 μm Millipore filters.

2.3. Viscometry

Solution viscosity measurements were carried out by Cannon–Ubbelohde viscometers: size 25, no. 115 for the viscosity range of 0.3–1.2 cSt, and size 50, no. 777 for the viscosity range of 0.8–4.0 cSt. Viscometers were submerged in a thermostated water bath with a temperature control precision of 0.1°C. A digital stop watch provided the flow time with a resolution of 0.01 s. For each solution, 3–5 viscosity measurements were taken and average values were computed and reported.

2.4. Light scattering

The light scattering apparatus was from Malvern, model

Table 1
Solution properties of HPC in water

M_w (g/mol)	R_g (nm)	A_2 (cm^3 mol g^{-2})	D_0 (cm^2 s^{-1})	R_h (nm)	[η] (l/g)
58 000	49	0.003	$1.474 \pm 0.02 \times 10^{-7}$	18.9 ± 0.2	0.132 ± 0.01

4700, equipped with a 2 W argon-ion laser producing a gaussian beam with a wavelength of 514.5 nm. The correlator used was a Malvern model 7032 with 128 channels and a temporal resolution of 50 ns. The sample cells used were 10 mm quartz cells for dynamic light scattering measurements and large Burchard quartz cells for the molecular weight measurement. The hole in front of our photomultiplier tube was set at 150 μm for dynamic light scattering and 500 μm for static light scattering.

Static light scattering measurements were taken based on the Zimm–Debye equation [20]:

$$Kc_p/\Delta R_\theta = (1/M_w)[1 + q^2R_g^2/3 + \ldots] + 2A_2c_p + \ldots \quad (1)$$

where ΔR_θ is the excess Rayleigh ratio, c_p the polymer concentration in g/l, M_w the polymer molecular weight, q the magnitude of scattering wave vector, R_g the radius of gyration, A_2 the solution second virial coefficient and K the optical constant defined as

$$K = 2\pi^2 n_0(\mathrm{d}n/\mathrm{d}c_p)^2/\lambda^4 N_A, \quad (2)$$

where n_0 is the solvent refractive index, n the solution refractive index and λ the laser wavelength. The solution refractive index increment was measured by a Dawn Optilab interferometric refractometer at 30°C, and determined to be $\mathrm{d}n/\mathrm{d}c_p = 0.164 \times 10^{-3}$ l/g.

Dynamic light scattering measurements were carried out to determine the translational diffusion coefficients and associated hydrodynamic radius of polymer chain. The measured light intensity correlation function, $g^{(2)}(\tau)$, is related to the electric field correlation function, $g^{(1)}(\tau)$, as follows [20]:

$$g^{(2)}(\tau) = 1 + \beta[g^{(1)}(\tau)]^2 \quad (3)$$

where β is the coherence factor and τ the delay time. The normalized light intensity autocorrelation function $g^{(1)}(\tau)$ can be written as the Laplace transform of the distribution of relaxation rate Γ as

$$g^{(1)}(\tau) = \int G(\Gamma)\exp(-\Gamma\tau)\,\mathrm{d}\Gamma. \quad (4)$$

To account for the multi-exponential decay of $g^{(1)}(\tau)$, we applied the cumulant fit to the electric field correlation function $g^{(1)}(\tau)$ as

$$\ln[g^{(1)}(\tau)] = -\bar{\Gamma}\tau + \mu_2\tau^2/2 - \mu_3\tau^3/6 + \cdots \quad (5)$$

where $\bar{\Gamma}$ is the mean decay rate and μ_i the ith moment. The mean value of the apparent diffusion coefficient $D_{app}(q) = \bar{\Gamma}/q^2$ is determined as a function of the scattering wave vector q in the limit of small q as

$$D_{app} = D_{CM}(1 + Cq^2R_g^2 + \cdots). \quad (6)$$

Here C is a dimensionless parameter depending on structure and polydispersity, R_g the radius of gyration of complex or HPC chain in solution, and D_{CM} the translational diffusion coefficient. The concentration dependence of D_{CM} can be written in the limit of small c_p as

$$D_{CM} = D_0(1 + k_Dc_p + \cdots), \quad (7)$$

where D_0 is the infinitely dilute diffusion coefficient, and k_D is related to the second virial coefficient A_2 as

$$k_D = 2A_2M - k_f - \bar{V}_2. \quad (8)$$

k_f is the concentration-dependence parameter of the friction coefficient and $\bar{V}_2$ is the partial specific volume of the polymer. The hydrodynamic radius R_h can be calculated from D_0 by applying the Stoke–Einstein relation as

$$R_h = k_BT/6\pi\eta_sD_0. \quad (9)$$

2.5. CMC and CAC measurements

The critical micelle concentration (CMC) and the critical aggregation concentration (CAC) were determined by two methods: surface tension measurement and conductivity measurement. The surface tension of the HTAB/water binary solution was measured by using a KRUSS (model K10T) tensiometer and the solution conductivity was measured using an Orion (model 160) conductivity meter. Both measurements were carried out at 30°C.

3. Results and discussion

3.1. Binary systems

The viscosity of dilute HPC solution was measured as a function of polymer concentration, and the intrinsic viscosity determined, by extrapolation of the reduced specific viscosity, $[\eta] = \lim_{c_p\to 0}\eta_{sp}/c_p$, to be $[\eta] = 0.13$ l/g. The corresponding overlap concentration $c^* = 1/[\eta]$ therefore was estimated to be 7.6 g/l. The hydrodynamic radius of the HPC chain was measured by dynamic light scattering as described above, and found to be 18.9 nm. The weight-average molecular weight M_w and the radius of gyration were determined from static light scattering measurements and found to be 58 100 and 49.5 nm, respectively, and the second virial coefficient $A_2 = 0.003$ cm^3 mol g^{-1}. These results are consistent with published data on HPC solutions [21]. Table 1 summarizes the solution properties.

For the HTAB solution, the CMC was determined from both surface tension and conductivity measurements. The

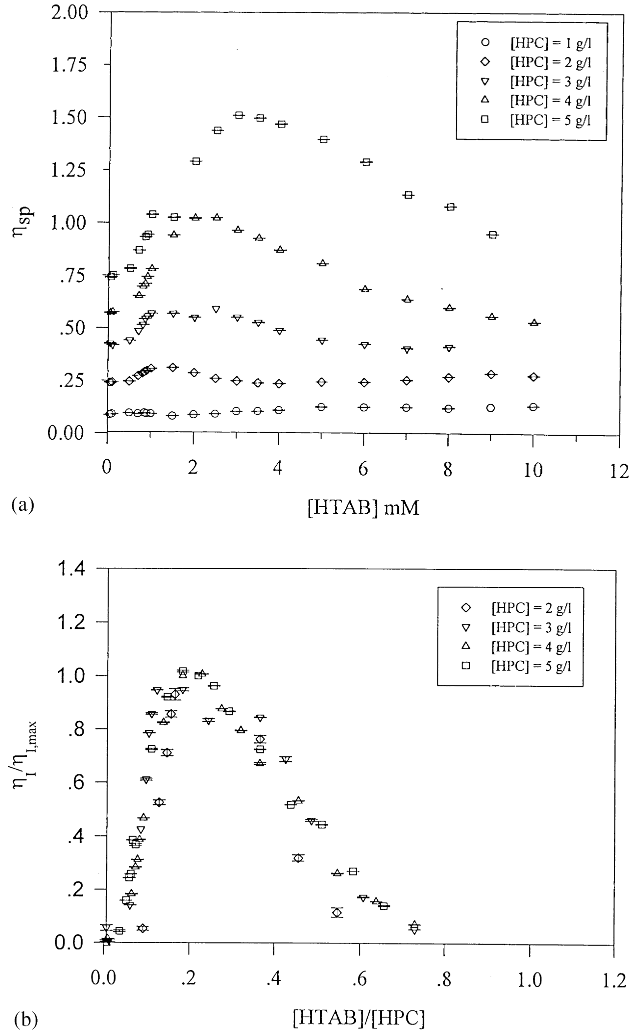

Fig. 1. The dependence of viscometric functions on polymer concentration [HPC] in the HPC/HTAB/water ternary system at 30°C: (a) specific viscosity vs. [HTAB] as a function of [HPC] (1, 2, 3, 4 and 5 g/l); (b) normalized interaction viscosity $\eta_I/\eta_{I,\max}$ vs. [HTAB]/[HPC] as a function of [HPC] (2, 3, 4 and 5 g/l). η_I is defined as $\eta_I = \eta - \eta_s - \eta_p$ where $\eta_{I,\max}$ is the maximum value of η_I, η_s is the solvent viscosity and η_p is the excess viscosity due to polymer in the HPC/water binary system.

CMC determined from these two plots was found to be 0.85 mM, comparable with previously published results [16,22–24].

3.2. Ternary systems

Fig. 1(a) shows the HPC/HTAB/water solution specific viscosity as a function of surfactant concentration at various polymer concentrations (1–5 g/l). The solution specific viscosities were measured 3–5 times at 30°C and average values were taken; error bars indicate that the combined uncertainties and experimental errors are within a few percent. The general trend apparent in Fig. 1(a) is that the specific viscosity increases initially at small HTAB concentrations to a maximum value, beyond which, at higher surfactant concentrations, there is a gradual decrease. The initial increase can be attributed to the increase in HPC chain size due to the electrostatic repulsion between micelles bound to the polymer chain. The chain expansion continues until the number of bound micelles reaches its maximum value, which we assume occurs where the specific viscosity also exhibits a maximum. The subsequent reduction in the solution viscosity as more micelles were added can be attributed to electrostatic screening due to the excess Br^- ions. We note that the amount of surfactant needed for maximum binding depends on the polymer concentration. In Table 2, we tabulate the corresponding HTAB concentrations, $[\text{HTAB}]_{\max}$, as a function of [HPC]. We see that $[\text{HTAB}]_{\max}$ increases from 0.18 to 1.10 g/l as the HPC concentration is varied from 1.0 to 5.0 g/l. This reflects that, as more HPC chains are available, a larger amount of HTAB is required for maximum binding. We find that, for all five HPC concentrations, the ratio $[\text{HTAB}]_{\max}/[\text{HPC}]$ is approximately constant at a value 0.18. Assuming that the ratio $[\text{HTAB}]_{\max}/[\text{HPC}]$ is equivalent to the number of bound surfactant molecules per polymer chain and using the known molecular weights for HPC and HTAB, we deduce that each chain interacts, on average, with 28 surfactant molecules.

Fig. 1(b) shows the plot of $\eta_I/\eta_{I,\max}$ vs. [HTAB]/[HPC] at various HPC concentrations, where η_I is the interaction viscosity defined as the excess of the ternary solution viscosity over that computed from the sum of the individual contributions of solvent, HPC, and HTAB:

$$\eta_I = \eta - \eta_s - \delta\eta_p - \delta\eta_{\text{surf}} \tag{10}$$

where η is the ternary solution viscosity, η_s the solvent viscosity, $\delta\eta_p$ the excess viscosity due to polymer alone in the binary polymer/water system, and $\delta\eta_{\text{surf}}$ the excess viscosity due to surfactant alone in the binary surfactant/water system. Thus, η_I reflects the interaction between the polymer and the surfactant in the ternary system. If no interaction occurs, between polymer and surfactant in the ternary

Table 2
HTAB concentrations at the maximum binding of CTAB micelles onto a HPC chain at various polymer concentrations

[HPC] (g/l)	$[\text{HTAB}]_{\text{viscosity}}$ (g/l)	$[\text{HTAB}]_{\text{Rh}}$ (g/l)	$[\text{HTAB}]_{\text{viscosity}}/[\text{HPC}]$	$[\text{HTAB}]_{\text{Rh}}/[\text{HPC}]$
1.0	0.18	0.22	0.18	0.22
2.0	0.36	–	0.18	–
3.0	0.55	0.55	0.18	0.18
4.0	0.73	–	0.18	–
5.0	1.10	0.87	0.22	0.17

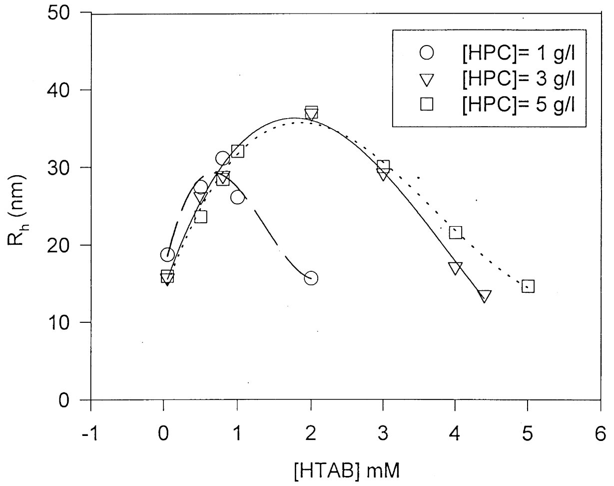

Fig. 2. The dependence of the center of mass hydrodynamic radius R_h on [HTAB] and [HPC] in the HPC/HTAB/water systems at 30°C.

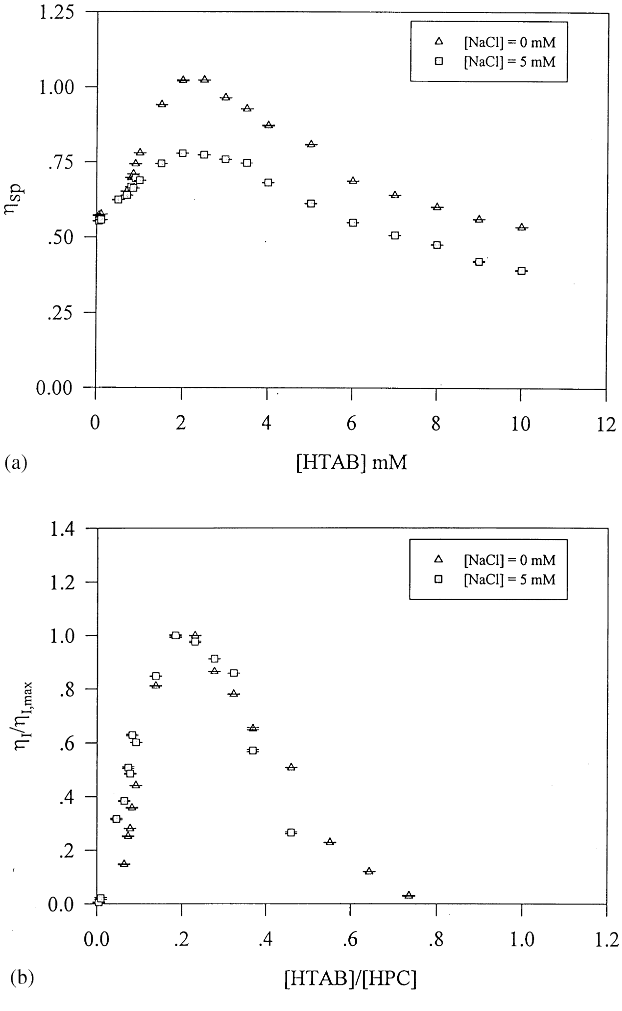

Fig. 3. The dependence of viscometric functions on NaCl concentration in the HPC/HTAB/water ternary system at 30°C and with [HPC] = 4 g/l : (a) specific viscosity vs. [HTAB] as a function of [NaCl] (0 and 5 mM); (b) normalized interaction viscosity $\eta_I/\eta_{I,max}$ vs. [HTAB]/[HPC] as a function of [NaCl] (0 and 5 mM).

system, such that polymer chains and surfactant micelles exist freely and contributes independently to the viscosity, we expect that η_I should be zero. From Fig. 1(b), it can be seen that, for each HPC concentration, the function $\eta_I/\eta_{I,max}$ exhibits common scaling when plotted as a function of the ratio [HTAB]/[HPC] with a maximum near [HTAB]/[HPC] = 0.18. By ‘common scaling’ we mean that for a given [HTAB]/[HPC] ratio, i.e. for a specified number of surfactant micelles bound per chain, the ratio of the interaction viscosity relative to its value at maximum binding is the same, regardless of the number of polymer chains in the solution.

The physical origin of this scaling is not clear, but presumably reflects the combined effect of charge density of the complex and screening of the electrostatic interaction on the viscosity. Some insight can be obtained by realizing that we are in the dilute solution regime, and hence may assume that the viscosity of polymer, surfactant, and ternary solutions follows the Einstein law:

$$\eta_p = \eta_s(1 + 2.5 n_p V_{hp}) \tag{11}$$

$$\eta_{surf} = \eta_s(1 + 2.5 n_{mic} V_{hmic}) \tag{12}$$

$$\eta = \eta_s(1 + 2.5 n'_c V_{hc} + 2.5 n'_p V_{hp} + 2.5 n'_{mic} V_{hmic}) \tag{13}$$

where n'_p and n'_c are the number concentrations of free polymer and complex, respectively, in the ternary solutions, and necessarily, $n'_p + n'_c = n_p$, the total number of HPC chains in the solution, V_{hp} and V_{hc} are the hydrodynamic volumes of free polymer and complex, n'_{mic} and V_{hmic} are the number concentration and hydrodynamic volume of free micelles in the ternary solution, which are present only after maximum binding. Within this approximation, from Eq. (10), we obtain:

$$\eta_I = 2.5\eta_s(n'_c V_{hc} + (n'_p - n_p)V_{hp} + (n'_{mic} - n_{mic})V_{hmic}) \tag{14}$$

Since the experiment tells us that the surfactant contribution is negligible, and we have that $(n'_p - n_p) = n'_c$, we can further simplify to:

$$\eta_I = 2.5\eta_s n'_c (V_{hc} - V_{hp}) \tag{15}$$

Now, realizing that, at $[\mathrm{HTAB}]_{max}/[\mathrm{HPC}]$, $n'_c = n_p$, we obtain:

$$\eta_I/\eta_{I,max} = (n'_c/n_p)(V_{hc} - V_{hp})/(V_{hc}^{max} - V_{hp}) \tag{16}$$

where V_{hc}^{max} corresponds to the values of V_{hc} at maximum binding. Essentially, here we assume random binding of micelles to the HPC chains. Eq. (16) suggests that the universal scaling in $\eta_I/\eta_{I,max}$ derives from the dual fact that the fraction of chains which form complex and the degree of swelling of the complex hydrodynamic volume relative to that of the free polymer are functions only of the ratio [HTAB]/[HPC].

The above interpretation is supported by the DLS measurements of the apparent hydrodynamic radius R_h vs. [HTAB] at various polymer concentrations, shown in Fig. 2. We can see that R_h rises to a maximum and then decreases as more surfactant molecules are added to the solution. This behavior is qualitatively consistent with the viscometric data of Fig. 1(a), supporting the interpretation that, as more micelles bind to an HPC chain, chain expansion occurs due to electrostatic interaction, followed by a chain contraction due to electrostatic screening. We point out that the maximum binding point in R_h occurs at [HTAB] = 0.02, 0.05 and 0.84 g/l for polymer concentrations of 1.0, 3.0 and 5.0 g/l, respectively. The corresponding values of the ratio $[\mathrm{HTAB}]_{\max}/[\mathrm{HPC}]$ compare favorably with those determined by the viscometric measurements as shown in Table 2.

The effect of ionic strength on the viscosity of the ternary solutions was studied. Fig. 3(a) shows the specific viscosity as a function of surfactant concentration for the two levels of added salt: 0 and 5 mM of NaCl. The specific viscosity at any particular [HTAB] level for the salt added solution is smaller than that of the salt free system. This supports that the observed changes in viscosity are electrostatic in nature. With salt added, Cl^- ions screen the electrostatic repulsion between micelles on a given chain, resulting in chain size reduction and hence a decrease in viscosity. Fig. 3(b) shows the normalized interaction viscosity, $\eta_I/\eta_{I,\max}$, evaluated from Eq. (10) at the two ionic strengths: 0 and 5 mM of NaCl. Both curves show scaling behavior with respect to [HTAB]/[HPC] identical to that of Fig. 1(b). The scaling function is thus found to be independent of the solution ionic strength, further consistent with the idea that the effect seen in viscosity and hydrodynamic radius originates from the effect of charge interactions between bound micelles on the HPC chain.

4. Conclusions

Our observations show convincingly that a binding interaction occurs between HTAB surfactant micelles and HPC chains. Above the critical micelle concentration, charged micelles bind to the non-ionic polymer chains resulting in a chain expansion and consequent rise in viscosity. Above the maximum binding surfactant concentration, excess micelles exist freely in the ternary solutions, and their counter-ions contribute to electrostatic screening between the bound micelles on a given polymer chain. The effect of electrostatic interaction on the viscosity of the polymer/surfactant/water ternary system can be described by a common scaling of the normalized interaction viscosity as a function of the surfactant/polymer concentration ratio, independent of solution ionic strength.

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