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Micellar Morphology in the Presence of Salts and Organic Additives

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Viscometry under Newtonian flow conditions has been used to study morphological changes in 0.3 M sodium dodecyl sulfate (SDS) micellar solutions in the presence of aliphatic amines (*n*-heptyl, C₇NH₂, or *n*-octyl, C₈NH₂) and different inorganic (MCl, M = Li, Na, K, or Rb; and NaX, X = Cl, Br, or SCN), organic salts (NaY, Y = salicylate (Sal), benzoate (Ben), anthranilate (An), or tosylate (Tos)), symmetrical quaternary bromides (R₄NBr, R = H, CH₃, C₂H₅, *n*-C₃H₇, *n*-C₄H₉, or *n*-C₅H₁₁) or tetrabutylphosphonium bromide, (n-C₄H₉)₄PBr, at 30 °C. Addition of amines with or without organic/inorganic salts causes the usual viscosity increase until the phase boundary of the L₁ phase is reached. However, in the presence of quaternary bromides, viscosity–[amine] profiles show an increase, a plateau, and slower decrease regions. The appearance of these regions has been found to be dependent on the chain length of the R-part and [quaternary bromide]. These effects have been discussed on the basis of solubilization of aliphatic amines in a micellar palisade layer or micellar exterior and their possible effects on the packing parameter and micellar morphology.

Introduction

Micellar association in the presence of organic additives has been extensively examined during the past 2 decades.^{1–8} The exclusive attention given to these systems is the result of the fact that surfactants are always used in combination with one or more additives simultaneously for their scientific, experimental, industrial, and theoretical applications.^{9–12} Surfactant solutions have a general tendency to solubilize a certain amount of organic additives. The environment of solubilization of different additives in or around micelles can be correlated with their structural organizations and mutual interactions.^{13,14} There are many factors, including the nature and the concentration of the additive(s), which determine the shape of the micelle. The packing parameter, $R_p (= V/A_0l_c)$, V_c being the volume of the hydrocarbon part of the surfactant molecule, l_c its length, and A_0 the surface area for surfactant molecule), for micellar morphology involves the nature of the head and tail groups of the surfactant.¹⁵ Surfactants with lesser head group areas tend to form larger, less curved, or even reversed structures. For ionic

surfactants, the above area shrinking effect may be achieved by addition of a counterion (salt) or a suitable organic additive (cosurfactant).

In the past 5–6 years we have been involved in studying the effect of salts and organic additives in ionic micellar solutions present individually or simultaneously by viscosity and small-angle neutron scattering (SANS) techniques.^{5–8,16–22} During the studies it has been observed that viscosity behavior changed drastically in cases of the simultaneous presence of salts and organic additives compared to the behavior of individual addition of salts and organics. Further, it was concluded that the location/partitioning of the additive at different regions of the micelle played a crucial role toward its effect on micellar structure and growth. SANS data showed that unidimensional growth took place when quaternary ammonium salts or aliphatic amines were added to sodium dodecyl sulfate (SDS) micelles.^{18,21} With cetylpyridinium bromide ((CP)Br), an opposite viscosity behavior to that of simple inorganic salts was found when aromatic hydrocarbons were added in the presence of tetra-*n*-butylammonium bromide (Bu₄NBr). With (CP)Br, it is obvious that Bu₄N⁺ would behave as co-ion and would remain in the bulk solvent to affect the micellar structure (by changing the solubilization site of aromatic hydrocarbons).⁸ In the case of anionic SDS, it is possible that Bu₄N⁺ could exist near the micellar surface with one (or more) *n*-butyl chain(s) intercalated between the head groups (extension of palisade layer toward bulk water).^{21,23} On the other hand, *n*-alkylamines, due to their hydrophobic and polar char-

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acters, could also partition in the head group region of the SDS micelle.^{24,25} Therefore, it will be interesting to see the effect of the simultaneous presence of the above two classes of additives having a preference for similar solubilization sites.

Surfactant solutions containing spherical micelles are of an isotropic nature and of low viscosity.²⁶ The presence of reasonably long rod-shaped micelles in the solution imparts higher viscosity to the solution.¹¹ In our earlier studies, we have proposed that interfacial partitioning of organic additives causes micellar growth while interior solubilization produces *swollen* micelles.⁶ These two types of micelles impart different viscosity behavior to micellar solutions.

In view of the fact that viscosity is sensitive to the shape/size of the microscopic objects in a homogeneous suspension, one can expect the evolution of the micellar shape to be reflected in the viscosity variations. However, quantitative interpretation of the viscosity data is out of the scope of the present paper since it is undesirable to extract size information from simple viscosity data.

The purpose of the present study is, therefore, to see the viscosity behavior of SDS micellar solutions in the presence of a variety of counterions and co-ions present concurrently with an organic additive. The nature of these ions may influence the partitioning content of the organics at different solubilization regions with a concomitant change in the micellar growth (or viscosity).²⁷ With this viewpoint we report herein the viscometric studies performed on aqueous anionic SDS micellar solutions with aliphatic amines (C_7NH_2 or C_8NH_2) across their entire composition range (until the phase boundary of the L_1 phase is reached) in the presence of salts of different categories, viz., (i) (a) inorganic salts, NaCl, NaBr, NaSCN, LiCl, KCl, and RbCl; (b) organic salts, sodium salicylate (NaSal), sodium benzoate (NaBen), sodium *p*-toluene sulfonate (NaTos), and sodium anthranilate (NaAn); and (ii) symmetrical quaternary bromides, NH_4Br , tetramethylammonium bromide (Me_4NBr), tetraethylammonium bromide (Et_4NBr), tetra-*n*-propylammonium bromide (Pr_4NBr), tetra-*n*-butylammonium bromide (Bu_4NBr), tetra-*n*-butylphosphonium bromide (Bu_4PBr), and tetra-*n*-pentylammonium bromide (Am_4NBr).

Experimental Section

Sodium dodecyl sulfate (SDS), *n*-heptylamine (C_7NH_2) and *n*-octylamine (C_8NH_2) were the same as those used earlier.¹⁸ Inorganic salts, NaX ($X = Cl, Br, SCN$) and MCl ($M = Li, Na, K, Rb$) were E. Merck products and were further purified by ignition. Organic salts, sodium salicylate, sodium benzoate, sodium anthranilate, and sodium tosylate, were from Fluka (~99%). All the quaternary ammonium and phosphonium bromides were also from Fluka (~99%). The salts were dried for at least 72 h before use in a vacuum drying oven. The temperature during drying was maintained according to the thermal stability and fusion point of the salt. The water used to prepare the solutions was demineralized and double-distilled in an all-glass distillation apparatus. The specific conductivity of this water was in the range $(1-2) \times 10^{-6} \Omega^{-1} cm^{-1}$.

The sample solutions were prepared by taking requisite amounts of C_7NH_2/C_8NH_2 by disposable micropipets in standard volumetric flasks and making up the volumes with the stock solution (0.3 M SDS containing either a fixed concentration of salt or no salt). The SDS concentration was fixed (0.3 M) throughout the work. The samples were left for equilibration

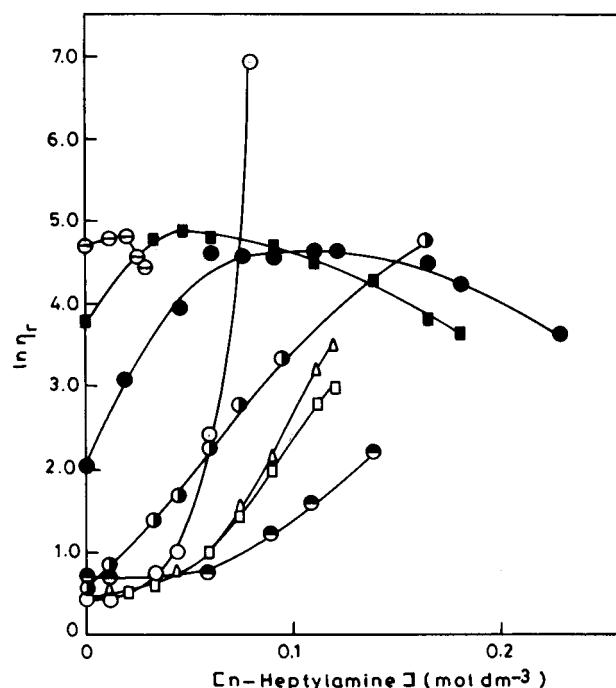


Figure 1. Relative viscosities (η_r) of 0.3 M SDS + 0.1 M R_4NBr micellar solutions as a function of added *n*-heptylamine (up to the solubility limit) at 30 °C: $R = H$ (\circ), CH_3 (Δ), C_2H_5 (\square), $n-C_3H_7$ (\odot), $n-C_4H_9$ (\bullet), $n-C_5H_{11}$ (\ominus), \blacksquare stands for tetra-*n*-butyl phosphonium bromide, $(n-C_4H_9)_4PBr$; \circ stands for without any salt.

(≈ 24 h). Prior to measurements, the Ubbelohde viscometer with the sample solution was kept thermostated at 30 ± 0.1 °C for at least 1 h to attain thermal equilibrium. The flasks and viscometer were kept properly stoppered and sealed during equilibration. At higher *n*-heptylamine/salt concentrations, the viscosities were dependent on the rate of flow. Viscosity measurements for such solutions under Newtonian flow conditions were performed as described elsewhere.¹⁸ No density corrections were made, since these were negligible.²⁸

Results and Discussion

It is known that micelles grow from globular to wormlike (rod-shaped) on addition of salts. Mukerjee²⁹ had proposed that an additive which is surface active to a hydrocarbon–water interface will be mainly solubilized at the head group region and will promote micellar growth. Amines, which may exist in protonated form, have been noticed to be more effective with anionic surfactants.³⁰ Further, we have found that the quaternary ammonium bromides produce micellar growth with SDS.²¹ The results embodied herein are discussed in light of these facts.

Figure 1 shows the viscosity variation of 0.3 M SDS micellar solutions (with or without 0.1 M quaternary ammonium or phosphonium bromides) on addition of C_7NH_2 . The viscosity remains almost constant when C_7NH_2 was added gradually (without salt), but a distinct rise in viscosity at higher concentrations of C_7NH_2 can be seen. Amines have been found to be solubilized in SDS micelles by electrostatic and hydrophobic effects with amine groups left on the micellar surface.²⁴ The SDS/ C_7NH_2 association will reduce surface area (A_0). Also, because of intercalation of the protonated C_7NH_2 , the alkyl part of C_7NH_2 would exist between two SDS monomeric

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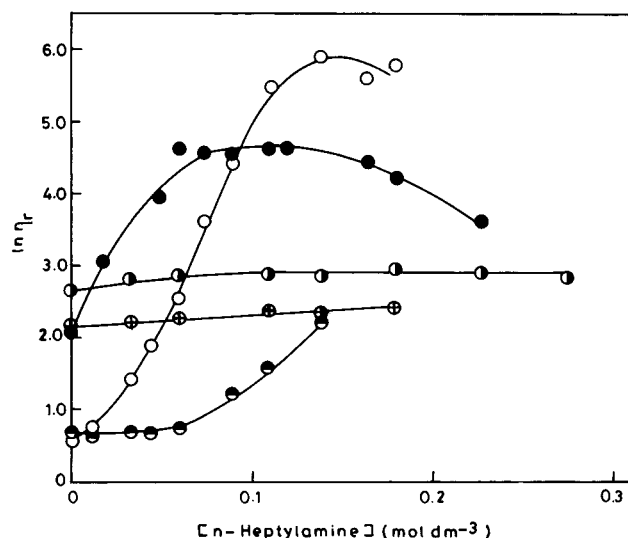


Figure 2. Relative viscosities (η_r) of 0.3 M SDS micellar solutions as a function of added *n*-heptylamine (up to the solubility limit) at various fixed concentrations (x) of $(n\text{-C}_4\text{H}_9)_4\text{NBr}$ at 30 °C: $x = 0.0$ (●), 0.05 (○), 0.1 (●), 0.2 (○), and 0.3 M (⊕).

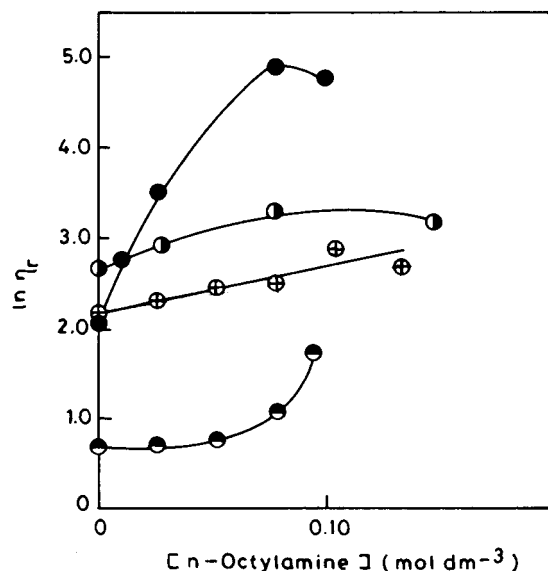


Figure 3. Relative viscosities (η_r) of 0.3 M SDS micellar solutions as a function of added *n*-octylamine (up to the solubility limit) at various fixed concentrations (x) of $(n\text{-C}_4\text{H}_9)_4\text{NBr}$ at 30 °C: $x = 0.0$ (●), 0.1 (●), 0.2 (○), and 0.3 M (⊕).

chains. This penetration will result in increasing the volume of the micellar core, which is equivalent to increasing the volume of the hydrophobic portion of the SDS molecule (V_h).³¹ This seems to result in an increase in R_p ,¹⁵ which is responsible for micellar growth, and an increase in viscosity of the 0.3 M SDS solution.

In the presence of NH_4Br , the viscosity increase was steeper than with other salts. This is possibly due to the fact that the NH_4^+ size (hydrated) is smaller than other R_4N^+ studied here. In this case the NH_4^+ screens out the negative charge of the SDS micelle, which is responsible for larger micelles and higher viscosities.³² In light of higher hydrated size of tetramethylammonium (Me_4N^+) and tetraethylammonium (Et_4N^+) ions the viscosity

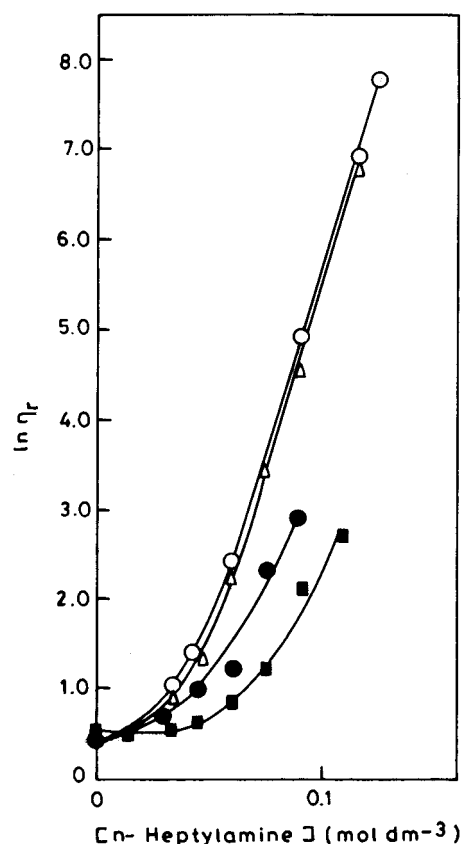


Figure 4. Relative viscosities (η_r) of 0.3 M SDS + 0.1 M MCl micellar solutions as a function of added *n*-heptylamine (up to the solubility limit) at 30 °C: $M = \text{Li}$ (■), Na (●), K (△), and Rb (○).

behavior with C_7NH_2 addition can be understood. The viscosity variation with tetra-*n*-propylammonium (Pr_4N^+) ion again shows a peculiar behavior which cannot be discussed in light of hydrated size terms as it has a bigger size but still has higher viscosities of the solutions than of Me_4N^+ or Et_4N^+ in the lower C_7NH_2 concentration range. Further, an increase in the alkyl part of R_4NBr showed a drastic change in viscosity variation. Such an organic ion having a number of hydrocarbon moieties has a large hydrophobic volume, and some of these four short alkyl chains may penetrate the micelle due to the hydrophobic effect. On the other side, the geometric requirement makes it infeasible for all four chains to penetrate into the micellar hydrocarbon region as proposed earlier.²¹ At least two chains should be located at the anionic micellar surface layer in contact with water, which forms an iceberg structure around the chains.²³ Generally two directions may be chosen for bending the alkyl chains: one is toward the water phase and the other penetrates the micellar hydrocarbon region. SANS results obtained on similar systems show an increase in the micellar aggregation number and confirm the above proposition.³³ In a separate SANS study it was also shown that micellar growth (and also an increase in the aggregation number) takes place in the presence of *n*-alkylamines in 0.3 M SDS, due to palisade solubilization of amines.¹⁸ Here also the initial viscosity increase of the 0.3 M SDS + 0.1 M R_4NBr (where $R > n\text{-C}_3\text{H}_7$) system on addition of C_7NH_2 may be due to the modification of the packing parameter with simultaneous micellar growth. In this situation one or more alkyl chain(s) of R_4NBr salts and an alkyl chain of C_7NH_2

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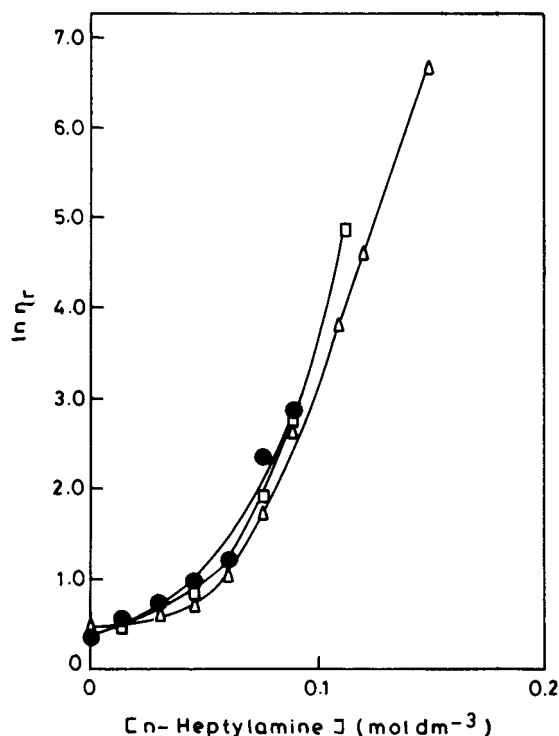


Figure 5. Relative viscosities (η_r) of 0.3 M SDS + 0.1 M NaX micellar solutions as a function of added *n*-heptylamine (up to the solubility limit) at 30 °C: X = Cl (●), Br (△), and SCN (□).

will be positioned at the same micellar solubilization site (between surfactant monomers) and be quickly saturated for such solubilization as the C_7NH_2 content is increased. At higher C_7NH_2 content, the solubilization of additional C_7NH_2 will be either inside the micelle (core region) or in the exterior of the micelle. Perusal of Figure 1 shows that with 0.1 M R_4NBr ($R = n\text{-}C_4H_9$ or higher) a constancy in viscosity is observed at fairly higher C_7NH_2 content. As mentioned earlier, C_7NH_2 is slightly protonated and has a hydrophilic NH_2 group; therefore, in the presence of anionic SDS micelles it will prefer the surface region and its core solubilization can be safely ruled out. As the palisade layer is saturated with alkyl chains of the symmetrical quaternary salts and a part of the C_7NH_2 content, the additional C_7NH_2 could be solubilized in the exterior region of SDS micelles, where the remaining alkyl chains of R_4NBr salts exist and may produce another temporary hydrophobic region around the micelle. This C_7NH_2 solubilization in the micelle exterior will not affect the micellar growth and the viscosity; therefore, will remain constant, as is indeed observed in Figure 1. Similar reasoning was put forth earlier to explain the effect of the addition of phenol on the viscosities of ionic micellar solutions.³⁴

Data in Figure 2 are self explanatory for the mechanism we have put forth for explaining the behavior in Figure 1. As one can see, that viscosity rises steeply as we move from a 0 to 0.05 M concentration of Bu_4NBr . In this case enough space is left in the micellar palisade layer after filling butyl chains of Bu_4NBr in it. At higher C_7NH_2 content a small plateau region is also observed, which could be an indication of saturation of the palisade region by the alkyl chains of C_7NH_2 as well as of Bu_4NBr . This effect is much more pronounced when $[Bu_4NBr]$ was increased to 0.1 M with a wider plateau region. On further

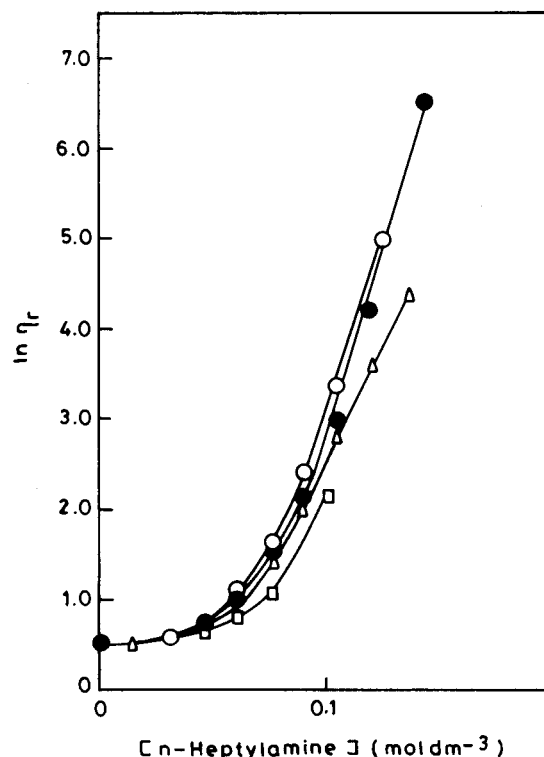


Figure 6. Relative viscosities (η_r) of 0.3 M SDS + 0.1 M NaY micellar solutions as a function of added *n*-heptylamine (up to the solubility limit) at 30 °C: Y = Sal (△), Ben (●), An (○), and Tos (□).

increase of the Bu_4NBr content to 0.2 or 0.3 M, no change in viscosity on addition of C_7NH_2 is found. This may be due to the fact that at higher Bu_4NBr concentration there is no room left for C_7NH_2 to solubilize in the palisade layer with the only choice being to solubilize in micellar exterior. This confirms earlier findings that palisade solubilization of an organic additive is most important to observe micellar growth in surfactant systems.^{6,8,34} Figure 3 shows the viscosity data with *n*-octylamine (C_8NH_2) additions. Here also only a little effect of alkyl chain length of amine could be seen, and the overall behavior was similar to that of C_7NH_2 addition. The difference observed is in the overall C_8NH_2 content solubilized in the system, which is obviously due to higher hydrophobic volume.

To compare the behavior of symmetrical quaternary counterions with simple inorganic counterions, studies were performed with alkali metal chlorides, and data are shown in Figure 4. Results of Figures 1 and 4 show that alkali metal counterions behave as NH_4^+ or lower members of the quaternary ammonium bromides, with no plateau region. These ions exist (adsorbed) at the micellar surface and have no reason to occupy sites in the palisade layer.^{35–37} Therefore, all added C_7NH_2 would go inside the palisade layer with a continuous increase in viscosity. The magnitude of viscosity at constant salt and C_7NH_2 contents follows the order $Li^+ < Na^+ < K^+ < Rb^+$. Nguyen and Bertrand³⁷ observed that alkali metal ions and 1-pentanol have cooperative effects inducing the sphere–rod transition and follow the same order. This is in qualitative agreement with our observation. The effectiveness of counterions in facilitating micellar growth (viscosity increase) follows the order of decreasing hy-

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hydrated size or increasing ionic crystal radius. This order was also observed by Missel et al.³⁸

Figures 5 and 6 show the effect of a variety of co-ions on viscosity (micellar growth) behavior of 0.3 M SDS with continuous addition of C_7NH_2 . The shapes of the viscosity curves with various sodium halides are similar with a small difference in the viscosity at a higher $[C_7NH_2]$ region. This difference in viscosity must be caused by the effect of the co-ion species on the hydrophobic interaction in the micelle formation or the extent of destruction of the hydrogen bonded structure of water. However, this effect has little influence on the micellar growth as compared to the counterion. Huisman³⁹ determined the micelle molecular weight of SDS in the presence of 0.1 M sodium halides and found that the micellar molecular weight was dependent on the co-ion species of the added salt in the order $NaF > NaCl > NaBr > NaI$. Our viscosity results for NaCl and NaBr seem to follow the order. The general trend is that the content of C_7NH_2 needed for producing a steep rise in viscosity is nearly independent of the hydrated anion size, which again confirms that alkali metal counterions and C_7NH_2 exist at different regions and co-ions have little effect on micellar growth. Variation of organic co-ions (Figure 6) also shows an effect similar to that of the inorganic co-ions (Figure 5). This means

that the small hydrophobic volumes which these organic co-ions have do not influence the partitioning of Na^+ and C_7NH_2 near the micellar surface and have very little contribution to the overall viscosity of the system. However, whatever difference in behavior is observed, it is mainly due to the co-ion effect on hydrophobic interaction, which can influence the micellar growth or viscosity.

The mechanism of the organic additives in the presence of salts is not fully understood. As indicated in the beginning, the purpose of this paper is to use viscosity data to study the effect of the simultaneous presence of salts and organic additives on micellar association structures (morphology). In such $SDS + R_4N^+$ systems, another local hydrophobic region, in addition to the micellar core, is produced which can be utilized for the solubilization of polar-organic compounds (drugs, pesticides, polyaromatic hydrocarbons, and pollutants, etc.). Currently, we are studying the association of micelles in the presence of salts and organic additives by SANS. It will shed more light on the phenomenon of the change of the solubilization site due to the presence of quaternary ammonium/phosphonium salts.

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