Micellar Association in Simultaneous Presence of Organic Salts/Additives

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ABSTRACT: Viscosity measurements under Newtonian flow conditions had been performed on cetyltrimethylammonium bromide (CTAB) aqueous solutions in the combined presence of sodium salts of aromatic acids (sodium salicylate, NaSal; sodium benzoate, NaBen; sodium anthranilate, NaAn) and organic additives (1-hexanol, C_6OH; *n*-hexylamine, C_6NH_2) at 30°C. On addition of C_6OH or C_6NH_2, the viscosity of 25 mM CTAB solution remained nearly constant without salt as well as with a lower salt concentration. This is due to low CTAB concentration which is not sufficient to produce structural changes in this concentration range of salts. However, as the salt concentration was increased further, the effect of C_6OH/C_6NH_2 addition was different with different salts: The viscosity first increased; then a decrease was observed with the former while with C_6NH_2 a decrease followed by constancy appeared in plots of relative viscosities (η_r) vs. organic additive concentrations. At further higher salt concentration, the magnitude of η_r was much higher. The viscosity increase is explained in terms of micellar growth and the decrease in terms of swollen micelle formation (due to interior solubilization of organic additive) or micellar disintegration (due to formation of water + additive pseudophase).

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KEY WORDS: CTAB, micelles, solubilization, swollen micelles, viscosity measurements.

Numerous studies have been carried out on the effect of salts (1–3) and organic additives (4–6) on the micellar structural transitions in aqueous medium. Recently, we reported a kind of "synergism" (e.g., significant increase in viscosity) in such systems when salts and organic additives are present concurrently in the micellar solutions (7–11). In these studies the nature of salts and additives was found to play a crucial role toward such synergisms.

Micelles of quaternary ammonium halides [e.g., cetyltrimethylammonium bromide (CTAB)] grow from spherical to rod-shaped on the addition of different counterions (12). Halide anions associate only moderately with surfactant headgroups, and micellar growth is gradual. However, with anions that associate strongly, such as aromatic salt anions (e.g., salicylate, Sal^-), rod-shaped micelles grow rapidly even at low surfactant and salt concentrations (13), and the solutions exhibit a remarkable viscosity increase (14,15).

Aqueous micelles are capable of solubilizing organic molecules with quite distinct polarities and degrees of hydrophobicity (16). The addition of different types of molecules leads to large deviations of packing parameters in micellar assembly (17). Many counterions and organic additives are strongly adsorbed at the micellar interface, and depending on the degree of penetration, this may change the mean distance between polar headgroups or may increase the volume of the micellar core. The aromatic acid counterions are most efficient with cationic micelles. ^1H nuclear magnetic resonance (NMR) studies have shown that the Sal^- anion orients in such a way that the negatively charged site (COO^- group) stands perpendicular to the CTAB micellar surface (18,19). This results in a large reduction of the net surface charge. Similar conclusions are also obtained from fluorescence measurements (20). Unlike homogeneous solvents, micelles possess a gamut of solubilization environments, ranging from the nonpolar hydrocarbon core of the micelle to the relatively polar micelle–water interface (21). The amount and the solubilization environment can play an important role in the resultant micellar morphology. Thus, solutes of different structural types may solubilize in distinct regions in or around the micelle. At the same time, solutes of similar hydrophobicities may compete for the similar environment inside the micelle. Mukerjee (22) proposed that an additive that is surface-active to a hydrocarbon–water interface would be solubilized mainly at the headgroup region and would promote micellar growth. In the study by Wormuth and Kaler (23), the hydrophilic ranking of organic additives (e.g., alcohols or amines) may be viewed in terms of the partitioning behavior between micellar and aqueous pseudo-phases. Amines were determined to be considerably more hydrophilic than alcohols. In our earlier studies we proposed that interfacial partitioning of organic additives causes micellar growth while interior solubilization produces swollen micelles (7) and that these two types of micelles impart different viscosity behavior to micellar solutions.

To improve their performance, surfactants are generally used in the presence of additives. Studies on surfactants and their mixtures with a variety of additives in aqueous solutions are therefore of interest regarding their technical applications (24–26). The presence of grown micelles imparts high viscosity to solutions, which might be of importance in industrial

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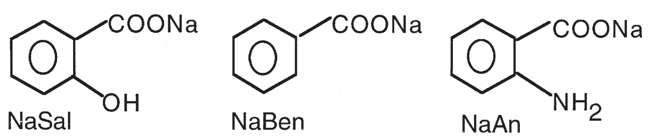

SCHEME 1

formulations as it enhances performance and customer appeal of the products. Control of viscosity of the formulations with additives can be achieved by varying the concentration and/or nature of additives. Solubilization of organics in aqueous surfactant solutions is useful in micellar-enhanced ultrafiltration (27,28). Here also, viscosity of the system could be useful to decide the size of the ultrafiltration membrane, as viscosity is used as a rough guide of the micelle size.

In view of the fact that viscosity is sensitive to 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 (29). Quantitative interpretation of the viscosity data is, however, outside 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 investigate the viscosity behavior of CTAB micellar solutions with representatives of aliphatic alcohol and amine families in the simultaneous presence of aromatic salt counterions (popular for strong micellar surface binding). This study provides interesting viscosity data for the systems in which the additives have preference for similar micellar environments. Very few such studies have so far been reported in the literature (10,11,30).

In the present communication, effects of addition of 1-hexanol and *n*-hexylamine on the viscosity behavior of CTAB micellar solutions have been seen in the presence and absence of different concentrations of aromatic acid salts (sodium salicylate, NaSal; sodium benzoate, NaBen; sodium anthranilate, NaAn: Scheme 1) at 30°C.

EXPERIMENTAL PROCEDURES

CTAB, 1-hexanol (C_6OH), *n*-hexylamine (C_6NH_2), NaSal, NaBen, and NaAn were the same as used earlier (7,10,31). The water used to prepare the solutions was demineralized and double-distilled in an all-glass distillation apparatus. The specific conductivity of the water was in the range of $1–2 \times 10^{-6}\ \Omega^{-1}\ \mathrm{cm}^{-1}$.

The samples were prepared by taking requisite volumes of C_6OH/C_6NH_2 by Hamilton microliter syringes (Bonaduz, Switzerland, supplied by CHROMATOPAK Analytical Instrumentation Pvt. Ltd., Bombay, India) in standard volumetric flasks and making up the volumes with the stock 25 mM (fixed) CTAB solution (containing either a fixed concentration of aromatic acid salt or no salt). The samples were left for equilibration (24 h). Prior to measurements, the Ubbelohde viscometer (fabricated in the laboratory) holding the sample solution was kept thermostated at 30 ± 0.1°C for at least 1 h to attain thermal equilibrium. The sample solutions in the flask and viscometer were kept properly stoppered and sealed during equilibration. At higher additive/salt concentrations, the viscosities were dependent on the rate of the flow. Viscosity measurements for such samples under Newtonian flow conditions were performed as described elsewhere (32). No density corrections were made, since these were negligible (33).

RESULTS AND DISCUSSION

Before discussing the results of CTAB/salt/organic additives, obviously we must discuss the CTAB/salt system and morphology of the aggregates in the solution. The zero-shear viscosity of such quaternary surfactants in the presence of salts in aqueous solutions showed two pronounced maxima when salts were added up to a fairly high concentration (13,34,35). It has been reported (14,15) that with an increase in NaSal concentration, micelles grow in length, begin to overlap, and entangle one another, leading to a large increase in viscosity. However, at low NaSal concentrations, the solutions contain slightly elongated micelles and their contribution to viscosity is comparatively small (36). We chose a low salt concentration range for our study of the effect of organic additives to avoid complexities of higher salt concentrations (e.g., double maxima), where micelles are not very different [although slightly elongated in the true sense (37)] from the globular nature of pure CTAB micelles (13). The organic counterions (Sal^-, Ben^-, An^-) used herein are believed to occupy the interfacial region with benzene ring partly incorporated into the micellar core of the cationic micelles (18,19,37). Two representative organic additives, 1-hexanol (alcohol) and *n*-hexylamine (amine), have been taken to see the effect of additive up to its solubility limit.

Effect of 1-hexanol. Figures 1–3 illustrate the interplay of relative viscosity, η_r (= η/η_0, where η and η_0 are the viscosities of the sample and solvent water, respectively) of 25 mM CTAB

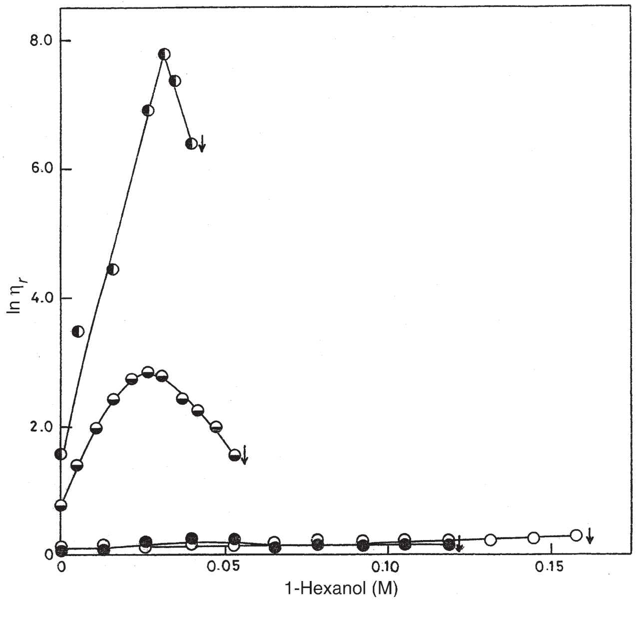

FIG. 1. Plots of relative viscosities (η_r) of 25 mM cetyltrimethylammonium bromide (CTAB) micellar solutions as a function of added 1-hexanol (up to the solubility limits indicated by arrows) at various fixed concentrations of NaSal at 30°C; 0 (○), 5 (●), 10 (◒), and 15 mM (◐).

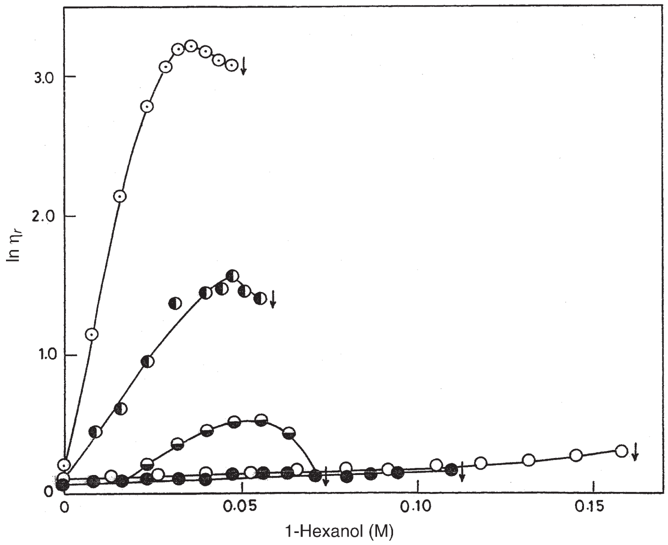

FIG. 2. Plots of relative viscosities (η_r) of 25 mM CTAB micellar solutions as a function of added 1-hexanol (up to the solubility limits indicated by arrows) at various fixed concentrations of NaBen at 30°C; 0 (○), 5 (●), 10 (◒), 15 (◐), and 25 mM (⊙). See Figure 1 for abbreviation.

with 1-hexanol addition at different fixed concentrations of salts (NaSal, NaBen, or NaAn) at 30°C. The addition of 1-hexanol to the CTAB solution (and also with 5 mM salt) has practically no effect on the viscosities as shown in Figure 1. This may be due to the fact that the small concentration of CTAB together with 1-hexanol, even in the presence of NaSal, has no significant effect on micellar morphology. The viscosity of the micellar solution containing short rod-shaped micelles with smaller axial ratio (~4) is not very different from that due to spherical micelles (38). Therefore, these short rods can hardly be detected in viscosity measurements. As the concentration of each salt is increased in the system, the viscosity patterns change with NaSal (Fig. 1) and NaBen (Fig. 2) while marginal changes occur with NaAn (Fig. 3) with increasing 1-hexanol concentration. This behavior may be due to different binding capacities of each counterion that would influence the packing parameters (17) to a different extent and hence to the micellar morphology and viscosity of the system.

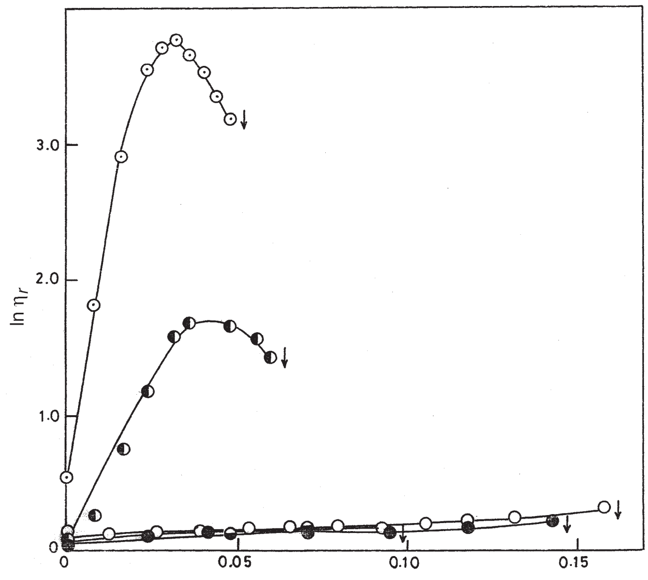

FIG. 3. Plots of relative viscosities (η_r) of 25 mM CTAB micellar solutions as a function of added 1-hexanol (up to the solubility limits indicated by arrows) at various fixed concentrations of NaAn at 30°C; 0 (○), 5 (●), 10 (◒), 15 (◐), and 25 mM (⊙). See Figure 1 for abbreviation.

Addition of 1-hexanol at higher NaSal content causes an increase followed by a decrease in viscosity. A similar type of viscosity behavior was observed earlier with inorganic salts (7,8). The difference we observe in the viscosity increase now occurs at quite low surfactant/salt concentrations. This is due to different binding abilities of the two kinds of counterions. The reasonable cause of the initial viscosity increase is the decrease in electrostatic repulsions (due to Sal$^-$) in addition to the increase in hydrophobic forces (due to intercalation of 1-hexanol between monomers of micelle). However, the observed viscosity decrease at higher 1-hexanol content is interesting. Owing to the –OH group, the alcohol has affinity to stay at the interfacial region. Therefore, by convention, the viscosity should not decrease as interfacial partitioning of an additive causes micellar growth (6,29,32, 39,40). Thus, another solubilization region inside or outside the micelle should be explored. There is a remote possibility of 1-hexanol being solubilized in the bulk aqueous phase. So only interior (micelle) partitioning is another possibility. If it is solubilized in the micellar interior, the elongation (grown micelle) of the micelle would start depleting, and another kind of micelle would be formed, i.e., swollen micelle, the solution of which could easily flow and cause a viscosity decrease: This indeed is observed in our case (Fig. 1). Similar types of viscosity behavior, except for magnitude, were observed with NaBen and NaAn (Figs. 2 and 3).

Effect of n-*hexylamine.* In comparison to 1-hexanol, the overall viscosity of *n*-hexylamine systems was distinctly lower (Fig. 4). Another point of difference was a slow increase in

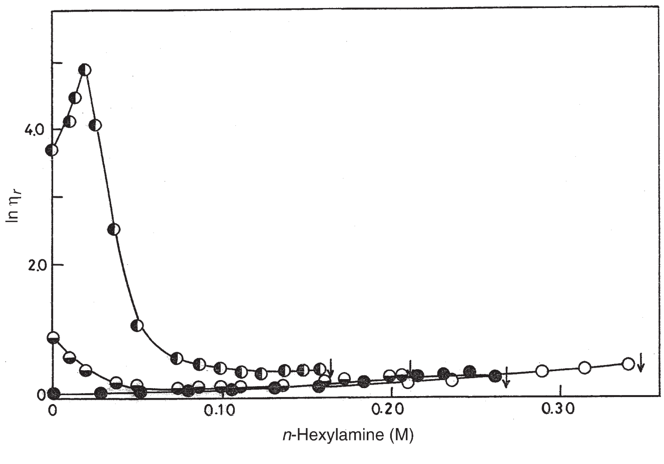

FIG. 4. Plots of relative viscosities (η_r) of 25 mM CTAB micellar solutions as a function of added *n*-hexylamine (up to the solubility limits indicated by arrows) at various fixed concentrations of NaSal at 30°C; 0 (○), 5 (●), 10 (◒), and 15 mM (◐). See Figure 1 for abbreviation.

viscosities with *n*-hexylamine concentration. One must remember that the –NH_2 group is more hydrophilic than the –OH group (23). This causes higher solubility of C_6NH_2 than C_6OH in aqueous solution, and one can expect partitioning of *n*-hexylamine in bulk aqueous and micellar phases in comparison to nearly total localization of 1-hexanol in the micellar phase. It was reported earlier that C_4–C_{10} NH_2 were solubilized in ionic micelles by electrostatic and hydrophobic effects with amine group left on the surface (41). Their partial dissociation into –NH_3^+ and OH^- (though feebly) may affect electrostatic interactions with the cationic headgroup. This effect may oppose micellar solubilization of the amine and micellar growth together with viscosity increase. In a lower NaSal concentration range, *n*-hexylamine addition causes either no change or a continuous decrease, followed by constancy in the viscosity (magnitude of η_r is low). This may be due to the fact that a low salt concentration would not be sufficient to neutralize the positive charge of the CTAB micelle with a net result in the repulsion of amine molecules from the micelle. Further, high hydrophilicity of C_6NH_2 would cause it to resist moving toward the micellar phase. These two interrelated factors cause C_6NH_2 to stay in the bulk solvent producing water plus C_6NH_2 pseudophases as observed by others for lower chain-length alcohols (42). This would influence the micellar structure by altering the organization of solvent molecules. Also, water plus C_6NH_2 would be a better solvent for the surfactant, resulting in withdrawal of monomers from the micelle and decrease of micellar size and viscosity. However, at higher NaSal concentration (15 mM), the viscosity increased in the initial concentration range of the amine addition. This may be due to the fact that at this NaSal concentration the micellar charge is possibly nearly (completely) neutralized and poses less (no) restriction on C_6NH_2 to go near the micellar surface and be solubilized in the interfacial region. This interfacial partitioning causes micellar growth and is responsible for viscosity increase, as indeed was observed (Fig. 4). However, continuous increase of C_6NH_2 at this NaSal concentration (15 mM) caused a drastic decline in viscosity, which seems to be due to the solvent effect of water + C_6NH_2, as discussed above. A similar viscosity decrease of micellar solutions in the presence of aliphatic hydrocarbons had been explained as core solubilization of hydrocarbons resulting in swollen micelles (8,43). This observed similarity of viscosity decrease by the addition of two types of organic additives (hydrocarbons and amines) known to partition at different sites (micellar interior and bulk aqueous phase) demands further discussion on the topic. In our case, the hydrophilic nature of C_6NH_2 would not permit us to propose solubilization of it in micellar interior, and the decrease can be looked upon as being attributable to dissolution of surfactant monomers in water + C_6NH_2 pseudophase. The experiments performed with NaBen and NaAn produced micellar solutions of very low viscosity, seemingly with no significant effect of C_6NH_2 addition on micellar morphology, which could be understood in the light of predominant partitioning of C_6NH_2 in the bulk solvent with these salts.

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