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ORIGINAL ARTICLE

The Clouding Phenomenon for Anionic Sodium Dodecyl Sulfate + Quaternary Bromides in Polar Nonaqueous-Water-Mixed Solvents

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Abstract Aqueous sodium dodecyl sulfate (SDS) solutions show clouding in the presence of tetra-*n*-butylammonium bromide (TBAB) and tetra-*n*-butylphosphonium bromide (TBPB). In this study, we report the effect of various volume percents of different polar nonaqueous solvents (acetonitrile, AN; dimethylsulfoxide, DMSO; methyl ethylene glycol ether, MC; formamide, FA; ethylene glycol, EG) on the clouding behavior of SDS + quaternary bromide (TBAB or TBPB) systems. The cloud point (CP) was found to decrease with initial increase in the volume percent of the above solvents in mixtures (with water). After a minimum in CP vs. volume percent plots, further increase in the volume percent caused an increase in the CP, followed by a near constant region. The data have been discussed on the basis of the effect of the above solvents on the two types of water present in the system: hydrated water and bulk water. As AN had shown a gradual decrease in the CP to a larger volume percent, it was chosen for detailed studies. Compared to TBAB, TBPB has diminished the effect of AN on CP increase due to its bigger size. The limited CMC data also run parallel to CP results.

Keywords Nonaqueous solvents · CP · CMC · SDS · Quaternary bromides

Introduction

Weighing the contributions of specific interactions between head groups, counterions and water in the interfacial regions of self-assemblies of surfactant molecules in aqueous solution that balance the hydrophobic effect and control the various transitions (e.g., structural and phase) of ionic micelles and biomembranes has proved arduous [1, 2]. Understanding the factors that control this self-assembly is central to a wide range of applications [3–5]. Despite their great significance, many important features of such self-assemblies have resisted theoretical explanation. Temperature is one of the important factors that dominate the formation of surfactant self-assemblies and, recently we have focused on several surprising and unexplained features of the temperature dependence of aqueous self-assembly of ionic surfactants in the presence of symmetrical quaternary bromides [6–11].

It is well known that below the Krafft point, ionic surfactants cannot form micelles [12]. In contrast, the aqueous micellar solutions of nonionic surfactants exhibit a lower consolute temperature, also called the cloud point (CP) [13]. The concentration dependence of the CP defines a phase boundary between the single and double phase systems, and the minimum temperature can be identified as the lowest consolute temperature. Above this temperature the solution separates into two phases: a concentrated phase containing most of the surfactant and another phase containing a surfactant concentration close to CMC. To explain this phenomenon, several mechanisms, including formation of mixed micelles, complexation or solubilization, have been proposed. Recent investigations have shown that the formation of the connected micellar network [14] or the strongly orientation-dependent interactions (H-bonds) between water and the surfactant heads

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[15] could be responsible for the CP behavior. Generally, the CP phenomenon can not happen in ionic surfactant systems because of the significant electrostatic repulsions between the charged self-assemblies. However, aqueous solutions of some ionic surfactants did exhibit the CP behavior in the presence of quaternary bromides having $R \geq C_4H_9$. The mechanism of the behavior is still an unexplained and challenging task in surfactant research [16–18].

Presently, two schools of thought regarding the appearance of clouding in ionic surfactant solutions are available : one says it is the removal of water by the counterions [7–9, 19] and the other says it is the geometric constrictions due to micellar growth [16, 17, 20]. The observations suggest the significant roles played by temperature and large hydrophobic counterions present in the vicinity of micellar head group region.

In most of their applications, surfactants with additives, rather than pure, are preferred due to the synergistic effect. The ingredients commonly present in industrial formulations strongly affect clouding behavior of surfactants [21]. Therefore, it is important to understand the magnitude and nature of such additive effects as well as the mechanism involved so that the system can be tailored to exhibit clouding behavior at desired temperatures. Although abundant literature is available on the effects of different additives on the CP behavior of nonionic surfactants solutions [22–24], those on the ionic surfactants are limited [7, 17, 20, 25–29]. In the case of anionic surfactant + quaternary salt systems, the effects of the nature of the counterion, the head group as well as the nature of the additives on the CP behavior were investigated [7, 9, 11, 29–33]. Clouding as well as micellization in nonaqueous polar solvents has, however, attracted less attention as compared to the case of water. Such studies can throw light on the CP phenomenon from the “solvophobic effect” point-of-view as opposed to the hydrophobic effect [34]. The term “solvophobic interaction” has been coined to describe aggregation in nonaqueous polar solvents, in analogy with “hydrophobic interactions”, responsible for the association of surfactants in water [35]. Therefore, it is of genuine interest to study the ionic surfactant clouding in polar nonaqueous solvents. Such studies may find use in the extraction of compounds through CP extraction methodologies [36, 37].

Our earlier studies on sodium dodecyl sulfate (SDS) + tetra-*n*-butylammonium bromide (TBAB) [7–9] have helped us to identify various SDS + TBAB combinations to see the effects of addition of acetonitrile (AN), dimethylsulfoxide (DMSO), methyl ethylene glycol ether (methyl cellosolve, MC), formamide (FA) and ethylene glycol (EG). CP measurements were also performed with SDS + tetra-*n*-butylphosphonium bromide (TBPB). A few CMC measurements have also been carried out in order to support the CP data.

Experimental Procedures

SDS (>99%, Fluka, Buchs, Switzerland), TBPB (99.5%, Fluka) and TBAB (>98%, Fluka) were used as received. All the nonaqueous solvents (AN, DMSO, MC, FA and EG) were of the highest purity grade available commercially and were used as obtained. Demineralized double-distilled water was used throughout.

Various SDS + quaternary bromide (TBAB or TBPB) samples were prepared by taking requisite amounts of SDS and the quaternary salt and making up the volumes with solvent (water or water + various specified volume percents of the nonaqueous solvent concerned).

The CP data were obtained by placing several Pyrex glass tubes, containing the sample solutions, into a temperature controlled bath. The temperature was ramped at the rate of 0.1 °C/min near the CP. Onset of turbidity (visual observation) was taken as the CP. However, the temperature was oscillated slowly through the CP until it was reproducible to ±0.1 °C.

Conductometry was used to determine the CMC and degree of counterion dissociation of the micelles (α) values [38]. The conductivity against SDS concentration plots showed typical profiles characterized by two linear regimes with different slopes, corresponding to the premicellar and postmicellar region, respectively, and where the break point is identified as CMC. Likewise the α values were calculated from the ratio between the slopes of these lines. The measurements were performed on an ELICO (type CM 82T) conductivity bridge equipped with platinized electrodes (cell constant = 1.02 cm^{-1}) at 30 ± 0.1 °C. The conductivity runs were carried out by progressively adding concentrated SDS stock solution into the thermostated mixed solvent (water or water + nonaqueous solvent).

Results and Discussion

Clouding in an SDS + TBAB System

It is well known that the micellar head group region is associated with a certain amount of water of hydration [39, 40]. Further, in ionic micellar solutions the counterion condensation plays very important role in deciding the effective charge on the micelle and hence its formation, structure, and mutual interaction. Pure SDS solution does not show any clouding but the presence of TBAB in the system is responsible for the clouding phenomenon (Fig. 1). The TBA^+ consists of four butyl chains in

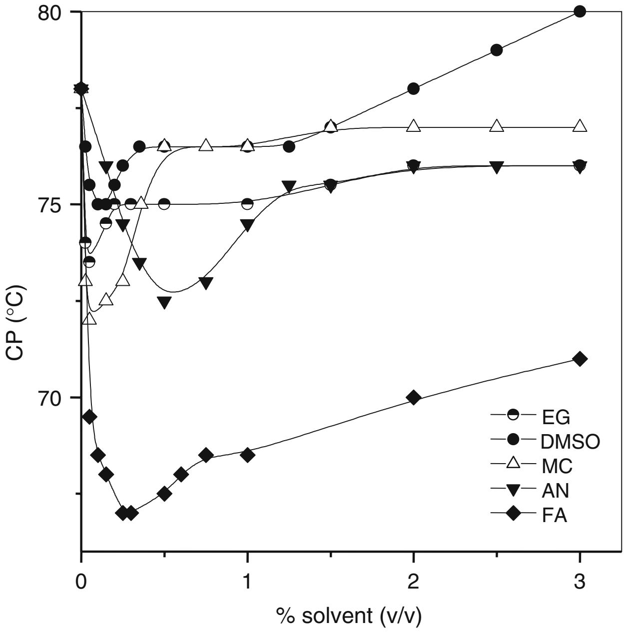

Fig. 1 Cloud point (°C) variation in the system 0.1 M SDS + 0.065 M TBAB with nonaqueous solvents

addition to the positive charge on the nitrogen atom. Hence the ion can interact with SDS micellar surface electrostatically as well as hydrophobically. In the present context, the butyl chains of TBA^+ may get embedded between monomers of the SDS micelle. As geometric constraint makes it difficult for all the four butyl chains to penetrate into the micelle core, two directions may be chosen for bending the butyl chains: one towards the water phase and the other towards the micellar core [7, 11, 19, 41]. The butyl chains towards the water phase may have the chance to interact with butyl chains of other counterions attached to other micelles. Consequently, micelles may experience closer contact, which may assist in replacing water of hydration from the head group region. Thus, the removal of water is attributed to the cloudiness of the SDS + TBAB system [11]. On the basis of the studies of CP variation with [SDS] or with [TBAB] [7–9], we have chosen 0.1 M SDS + 0.065 M TBAB system to study the effect of various volume percents of nonaqueous solvents on the CP (in mixed solvents).

Clouding in an SDS + TBAB System in Mixed Solvents

Figure 1 shows the variation of CP with volume percent of nonaqueous solvents present with water. A well defined minimum can be seen in all cases followed by an increase in CP at slightly higher volume percents of the nonaqueous solvents. Before the minimum (Fig. 1), the rate of decrease in CP with volume percent is different for different solvents. The decrease in CP seems to be dependent on the accumulation of the solvent at micelle-water interface. Due to this accumulation some of the water of hydration is replaced by the nonaqueous solvents. This results in a less hydrated micelle which therefore, requires a lower temperature to show clouding. This indeed is observed. The effect of the addition of nonaqueous solvents depends on how they change the water structure and micelle structure [42]. Although each solvent brings forward the clouding (before the minimum), the reasons are quite different. In the case of MC, the interaction consists of the destruction of the H-bonding of the original water near the micellar head group region and formation of new H-bonds between water and MC with a simultaneous depletion of micelle hydration and a decrease in the CP. The bringing forward of the CP in the presence of AN can also be understood in terms of the formation of H-bonds between water and AN. However, AN shows more gradual decrease in CP than MC (Fig. 1). This is probably due to the higher molar volume of MC (Table 1) than AN and, therefore, it could be more efficient in removing water from the head group region. It has been reported that EG alone or mixed with water stabilizes proteins against denaturation (increased hydrophobic interactions) [43, 44]. The clouding phenomenon in the present case is also facilitated by EG addition before the minimum and hence CP variation can be understood in the light of the discussion above. The dielectric constant of FA is the highest among the solvents listed in Table 1. In an independent study, Singh et al. [45] concluded that the

Table 1 Physical parameters of the solvents used and their concentrations (%, v/v) at the lowest CP of 0.1 M SDS + 0.065 M TBAB

Organic solvent	Mol. formula	Mol. wt.	Density (g cm^{-3})[a]	Molar volume (cm^{-3})	Dielectric constant	% solvent (v/v) at the lowest CP
AN	CH_3CN	41.05	0.786	52.56	39.0	0.50
MC	$CH_3OCH_2CH_2OH$	76.10	0.968	79.02	35.0	0.05
DMSO	$(CH_3)_2SO$	78.13	1.101	71.09	45.0	0.10
EG	$HOCH_2CH_2OH$	62.07	1.109	55.82	37.7	0.05
FA	$HCONH_2$	45.04	1.133	39.75	109.0	0.25

[a] Weast RC (ed) (1977) CRC Handbook of physics and chemistry. CRC Press, Florida

CMC decreases for the same surfactant according to the increase in the dielectric constant of the medium. The presence of FA near micellar heads would decrease repulsion between similarly charged head groups allowing the predominance of the hydrophobic interaction and hence a decrease in CP. Similar types of argument were given to explain the CMC decrease in the presence of a low urea concentration [46]. Therefore, the decrease in the CP in the presence of FA is in consonance with the earlier studies. The DMSO addition behavior is similar to that of other solvents before the minimum although DMSO is known to form stoichiometric hydrates with water [47]. The content of DMSO in the system below the minimum is low and one can expect that hydrate formation at such low content would also be minimal. The removal of water from the micelle head group region seems dominant and responsible for the CP decrease (Fig. 1). At higher bulk phase concentrations, the solvents decrease the dielectric constant of the bulk phase. This would cause increased mutual repulsion among the SDS heads in the micelle thus producing more hydrated micelles and hence the CPs are observed at higher temperatures (Fig. 1). Based on the data shown in Fig. 1, we have chosen AN for further study because it provides a gradual variation of the CP up to a higher volume percent below the minimum.

Figure 2 shows the variation of CP with volume percent of AN for x M SDS + 0.05 M TBAB system. From the perusal of CP data it is clear that as we increase [SDS] in the system the CP decreasing/increasing effect is more pronounced. Since addition of nonaqueous polar solvents affects the water as well as micelle structures it is expected that the surfactant content in the overall system would affect the interaction with AN and so the CP. At low concentrations of SDS, TBAB content is comparatively higher. As [TBAB] affects CP significantly [30], the effect of the AN content in the system seems less important. This means at higher [SDS], the role of AN is significant as the system has more micelle-bound water which is effectively replaced by AN molecules.

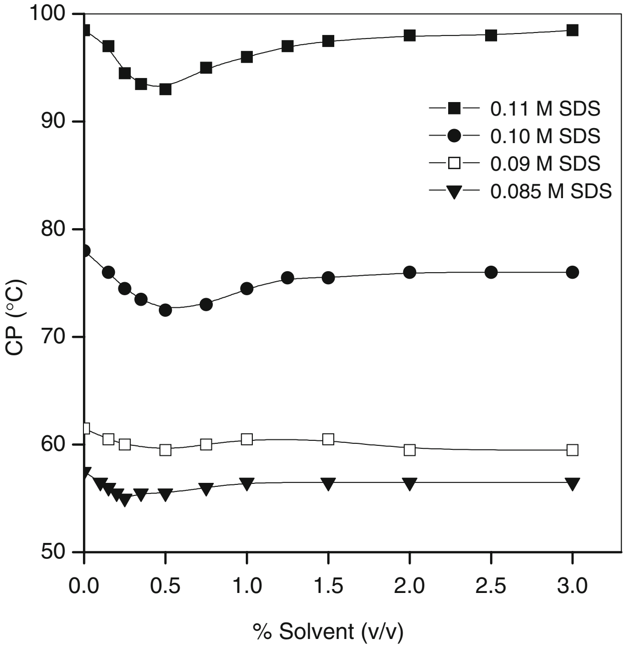

Fig. 2 Cloud point (°C) variation in the system x M SDS + 0.065 M TBAB with AN

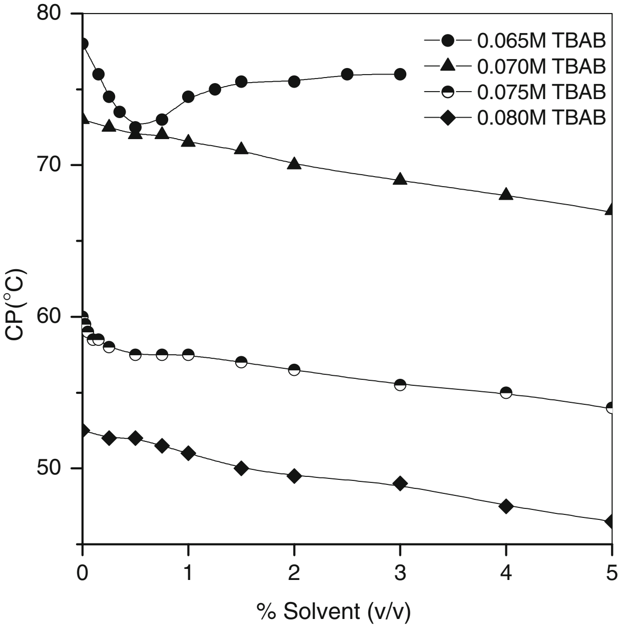

Fig. 3 Cloud point (°C) variation in the system 0.10 M SDS + y M TBAB with AN

To prove the above point we performed CP measurements with varying volume percents of AN in systems containing different fixed concentrations of TBAB (Fig. 3). The CP decreasing effect of AN is present up to a higher volume percent for systems containing more TBAB. This suggests that in presence of higher [TBAB] one can expect more TBA^+ counterions near the micellar surface. As discussed above that more number of TBA^+ would replace more hydrated water. Thus, less hydrated water would now be available to interact with AN which is responsible for the CP decrease.

Figure 4 shows the variation of the CP with the volume percent of AN for systems in which both SDS and TBAB were increased while the molar ratio (SDS/TBAB) was kept almost constant. The data show that these systems with higher contents of SDS and TBAB show a CP decrease at higher volume percent of AN. It can be understood by the fact that if we have more SDS, more number of micelles would be formed with total higher content of bound hydrated water. If it is so, higher AN is

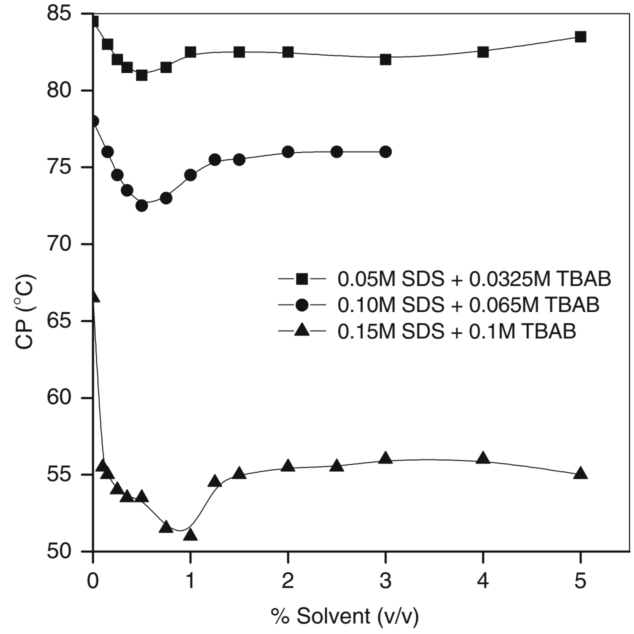

Fig. 4 Cloud point (°C) variation in the system *x* M SDS + *y* M TBAB with AN

needed to interact with this bound water and hence the CP decrease is expected to continue upto a higher AN content (i.e., the minimum would shift progressively to a higher AN content). This is what we observed in Fig. 4.

Similar experiments of CP measurements were also performed with an SDS + TBPB system (Fig. 5). The increase in both SDS and TBPB contents produced a CP decrease with volume percent of AN. Unlike Fig. 4, no minima were observed, but the trend of a higher content requirement of AN is clearly indicated if we consider the break points of nearly two straight lines for lower and higher AN contents. This difference in the presence of TBPB (compared to TBAB) may be due to the difference in sizes of TBP^+ and TBA^+. It was reported earlier also that a lower amount of TBPB is needed to observe clouding in comparison to TBAB [33]. Though the concentration ranges of the two sets of CP measurements (Figs. 4 and 5) are different, it is clearly possible to conclude that once the micellar head group region is crowded by quaternary counterions, the effect of the nonaqueous solvent to lower the CP is important because now less water remains there which allows more solvent to accumulate at the micellar interface. The CMC data for all the solvents at low volume percents are given in Table 2. The micelles formed in nonaqueous solvents (or mixed solvents with water) are similar in many respects to the micelles that are formed in water, although, micelle formation is not as favored in such solvents as in water for a given surfactant [48, 49]. However, the CMC data of Table 2 are obtained at comparatively much lower volume percents to get an insight into the CP behavior of the present systems before the minimum (Fig. 1). It is surprising to see that the CP decrease runs almost parallel to the CMC decrease in the presence of lower volume percents of the different solvents. This seems a very important result which hints at a modification of hydrophobic interactions at such a low volume percent. Later on the effect on water structure predominates and a regular CMC/CP increase is observed [34, 48, 49]. We can also see a weak dependence of α on concentration and the type of the solvent used. This is in

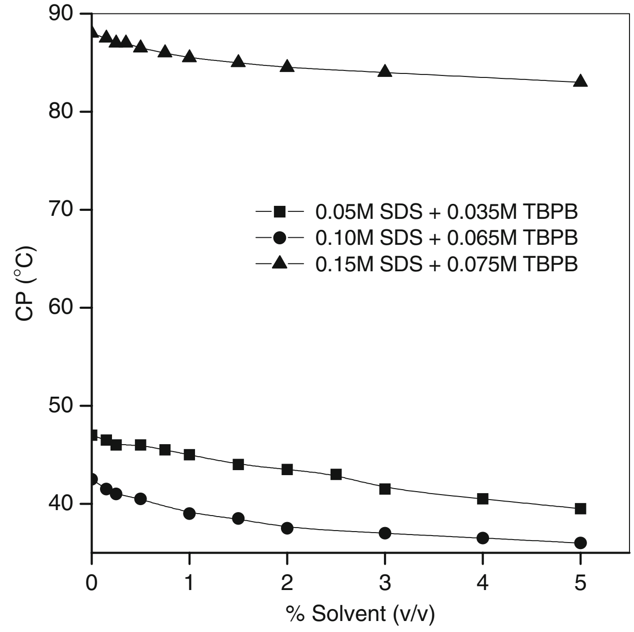

Fig. 5 Cloud point (°C) variation in the system *x* M SDS + *y* M TBPB with AN

Table 2 The CMC of SDS in water + non-aqueous solvents (without quaternary bromides) determined by conductometry at 30 °C

Organic solvent	% solvent (v/v)	CMC (mM)	Degree of dissociation (α)
AN	0	8.1	0.36
	0.25	7.7	0.39
	0.50	7.4	0.38
	1.0	7.6	0.42
	3.0	7.9	0.46
DMSO	0	8.1	0.36
	0.10	7.8	0.38
	0.50	7.9	0.40
	3.0	8.4	0.41
MC	0	8.1	0.36
	0.05	7.6	0.40
EG	0	8.1	0.36
	0.05	7.7	0.40

agreement with the literature results [35] which may be due to the preferential binding of both the surfactant and counterions to water than to the alternative polar nonaqueous solvent.

In conclusion, we can say that clouding that occurs in ionic surfactant sodium dodecyl sulfate solutions in the presence of quaternary bromides can be facilitated by certain volume percents of added polar nonaqueous solvents (mixed with water). The effect depends on the nature of the solvent used. Our limited CMC data also suggest that a new kind of behavior is expected if the micellization were to be studied in water containing low additions of the above solvents. One can expect unconventional results at low volume percents as were observed earlier in the case of low urea content [46].

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