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Physicochemical properties of mixed surfactant systems: sodium dodecyl benzene sulfonate with triton X 100

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Abstract The physicochemical properties of a mixed surfactant system were studied under various conditions. The surfactants were anionic sodium dodecyl benzene sulfonate and nonionic Triton X 100. Variation of specific conductivity with concentration was used to determine the critical micelle concentration of anionic as well as the mixed surfactants. Iodine solubilization method was used to determine the CMC of the nonionic surfactant. The interaction parameter between the surfactant molecules were calculated.

The wetting, foaming and detergent properties of mixed surfactant systems were studied. The variation of contact angle of the solution with teflon surface as a function of surfactant concentration was found to be a reasonably good method to determine the critical micelle concentration. Viscosity and cloud points were also determined. All these quantities are discussed.

Key words Mixed surfactant – wetting – foaming – detergency – triton X 100 – SDBS

Introduction

In continuation to our interest in the surfactant systems [1–5], both pure and mixed, we have studied in detail the various physicochemical properties of a mixed system consisting of anionic sodium dodecyl benzene sulfonate (SDBS) and nonionic polyoxyethylene (9.5) t-octylphenol (Triton X 100/TX 100) in water. The mixture of surfactants are important because it is but rare that single surfactant is present in industrial and in biological systems. The critical micelle concentration (CMC) of mixed surfactant systems have been studied in reasonable detail [6–13]. Variation of different properties of mixed surfactant systems have also been addressed [7]. It was observed that the interaction between surfactant monomers could be calculated by assuming the applicability of regular solution theory to these systems [14].

In the formulation of surfactant systems for practical applications like detergency [15,16], paints [17], enhanced oil recovery [18], the wetting, foaming, viscosity etc. properties are very crucial. Hence, a large number of publications are available in literature [17, 19–21] where such properties have been studied. Moreover linear alkylbenzene sulfonates are known as the work horse of the detergent industry [22–24] and have good foaming property. Nonionic surfactants are known to be less foaming. Generally an optimum condition is preferred. Therefore a mixture of anionic and nonionic surfactants would be very important to tune the surfactant mixture to the users' requirement.

Desai et al. [8] discussed the behaviour of a mixed surfactant system consisting of cationic and nonionic surfactants. They have noted that in general the mixed systems show negative deviation from ideality. Huang et al. [25] have discussed the mixed micelle consisting of anionic

sodium octanoate with ethoxylated alcohols. Rathman et al. [26] discussed the counter ion binding on mixed micelles of nonionic/anionic as well as nonionic/cationic surfactant systems. The sodium dodecyl sulfate–TX 100 system has been studied by Janczuk et al. [27].

In this paper, we are presenting various physicochemical properties of the chosen surfactant systems SDBS + TX 100 at different temperatures. The system was chosen rather arbitrarily because of our interest in anionic–nonionic surfactant systems and also for the fact that both SDBS and TX 100 are widely used and widely studied surfactants. Hence, mixtures of these two were intrinsically important and interesting to us. Moreover, in household detergent mixtures, anionic and nonionic surfactants are used. The critical micelle concentrations were determined. The wetting, viscosity, foaming, cloud point, detergency of these mixed surfactant systems were also studied. All these properties are discussed in the light of the known information till now.

Experimental section

The anionic surfactant sodium dodecyl benzene sulfonate (SDBS) and nonionic surfactant Triton X 100 (TX 100) were from Sigma Chemical Co. and were used without further purification. Water used was triply distilled with specific conductance $\sim 3 \times 10^{-6} \text{ S cm}^{-1}$. Teflon tape (Samson, India) was washed with chromic acid and copious quantities of water and dried before use. Chloroform (Qualigen, India) was used as received. Malachite green (CI 42000 Hopkins Williams, UK) was used as received.

The critical micelle concentration of SDBS and SDBS–TX 100 mixed systems were determined conductometrically. A Mullard (UK) conductivity bridge was used. A specific conductance – total concentration of the surfactants plot at constant temperature ($\pm 0.1^\circ \text{C}$) show break points which were taken as CMC of the system at the experimental temperature (Fig. not shown). The details of the method were discussed earlier [28]. The CMC of the TX 100 was determined by iodine solubilization method as discussed in an earlier paper [29]. The reproducibility in the CMC values is less than $\pm 1\%$.

The viscosity of the surfactant solution was determined with the help of an Ubbelohde viscometer at different temperatures. The cloud points of the surfactant solutions were determined visually by noting the temperature at which the turbidity appeared. We also noted the temperature at which turbidity disappeared on cooling. The average of these two values is the reported cloud point. The reproducibility in the cloud point was calculated to be less than $\pm 1\%$.

The wetting characteristics of the surfactant solutions were determined by the contact angle measurement. A contact θ meter, obtained as a gift from Department of Color Chemistry, University of Leeds, Leeds, UK, was used to measure the contact angle. Drops of liquid were made on the surface of the clean teflon tape with a syringe at least at 6 different places. The angles were determined at room temperature ($\sim 30^\circ \text{C}$) and average is being reported. The error in these values is less than $\pm 2\%$.

The foaming efficiency of the surfactant mixture was determined by the well known Ross and Miles [18, 30] method. In this method 200 ml of the thermostated surfactant solution from a separating funnel was allowed free fall of 90 cm through a tube of 1.5 cm internal diameter on 50 ml of same solution at the same temperature present in a 250 ml volumetric flask. The internal diameter of the orifice in the stopper of the separating funnel was 3.0 mm. The height of the foam in the tube was termed as initial foam height and was noted. The foaming efficiency was measured at least 3 times for each system and the average height is being reported here with an error of $\pm 1.8\%$.

Malachite green solution (0.1% w/v) was made in chloroform. This dye has been used in the spectrophotometric determination of trace amounts of P, Ti, Co, Ag in water, hair etc. [31–33]. Cotton fabric $3.8 \times 3.8 \text{ cm}^2$ was placed in 100 ml of this solution for one half hour under constant stirring. The fabric was then removed, dried in air and again immersed in 100 ml distilled water with constant stirring for 1 h, removed and dried before use. The cotton strip was then placed in 100 ml surfactant solution at the experimental temperature in a bottle with continuous shaking of the bottle. Absorbance of the solution was measured at regular time intervals at 428 nm by using a spectrophotometer (Shimadzu, UV 240).

Theory

Surfactant mixtures in solution, in general, form mixed micelle. The mixed micelles are expected to be in equilibrium with the monomer surfactant molecules. Assuming that the regular solution theory is applicable to a mixture of two surfactants A and B, it is possible to deduce the following two relations [14]

$$\frac{X_A^2 \ln(v C_m / X_A C_A)}{(1 - X_A)^2 \ln[(1 - v) C_m / (1 - X_A) C_B]} = 1, \quad (1)$$

$$\beta = \frac{\ln(v C_m / X_A C_A)}{(1 - X_A)^2}, \quad (2)$$

Table 1 Critical micelle concentration of SDBS–TX 100 mixed systems in aqueous solution at various temperatures

SDBS/TX 100	CMC [mM]				
	303 K	308 K	313 K	318 K	323 K
10:0	2.10 2.1 ^{a)}	2.25 ^{b)}	2.40	2.72	3.34
9:1	0.49	0.42	0.46	0.67	1.14
7:3	0.42	0.35	0.38	0.40	0.44
5:5	0.26 0.26 ^{a)}	0.31	0.37	0.41	0.42
3:7	0.37	0.35	0.36	0.42	0.47
1:9	0.38 0.33 ^{a)}	0.33	0.35	0.40	0.45
0:10	0.25 0.28 ^{a)}	0.23	0.22	0.22	0.20

^{a)} CMC obtained from contact angle measurements at $\sim 30^\circ\text{C}$ (see text).

^{b)} cf 2.14 mM at 303 and 2.40 mM at 313 K [54].

where C_A , C_B and C_m are the CMC of surfactants A, B and the mixture, respectively, v is the mole fraction of surfactant A in the solution and X_A is the mole fraction of the surfactant A in mixed micelle. By using Eq. (1) in an iterating computer programme where, besides X_A , all the quantities are known, the X_A can be computed. This gives the composition of the mixed micelle. Equation (2) is then used to compute the surfactant molecular interaction parameter β .

Results and discussion

In Table 1, the critical micelle concentration of SDBS, TX 100 and their various mixtures at different temperatures are presented. It is noted that for the ionic SDBS, the CMC continues to increase with increase in temperature whereas for nonionic TX 100, the CMC decreased with increase in temperature. This is a general phenomenon which indicates that though micellization is due to hydrophobic forces, for ionic surfactants the repulsive force is important [31–38]. Also hydrophilicity increases with increase in temperature. Therefore, higher concentration is required for the micellization to occur. However, for nonionic surfactants no such repulsive force exists and at higher temperature hydrophobicity increases due to the dehydration of ethylene oxide and hence the monomer molecules come near each other at lower concentration. The CMC decreases between 303–308 K before the rise of CMC with temperature. Generally such minimum is ob-

served for anionic surfactants and presence of TX 100 might have brought the minimum in the temperature range studied. We have checked the reproducibility of our data and they are within the limit of reproducibility as mentioned earlier.

As mentioned earlier in the theory section, the interaction parameter β between the monomers forming the mixed micelle can be calculated. We have not been able to compute the β quantity for all systems. The Eq. (1) did not converge for any value of X_A . That is we could not compute X_A and hence β . Most of the calculated values of β (–4 to +2) are negative indicating an attractive interaction between the surfactant molecules. Interestingly β is expected to be same over the concentration range of the surfactant at any particular temperature. However, we found that it was not so and even the sign changed. In some cases β becomes positive as the temperature or concentration ratio changes. This fact as well as our inability to get micelle composition by iteration procedure of Eq. (1) for some systems and hence corresponding β meant that the Rubingh's theory, though is useful in very many cases, is not probably applicable in all systems in all compositions. Motomura et al. [37], Puvvada et al. [7], and Haque et al. [38] have discussed this problem. The concentration dependent contradictory behaviour of β is difficult to explain [38]. The variation of β with composition has also been seen earlier [38–40].

The wetting property of the mixed surfactant solution was obtained by determining the contact angle of the solution with a clean teflon surface. It was observed that the wetting capacity of the mixed system is actually lower than the single surfactant when SDBS and TX 100 were mixed at various ratios, the concentration of each surfactant solution being 1.5 mM. However with SDBS (1.8 mM) and TX 100 (1.5 mM), the contact angle increases indicating less wetting capacity (Fig. 1) at room temperature. We postulate that the wetting characteristic of the solution depends on the amount of each of the component as well as the ratio of these components [28]. This is because surface activity of pure components are not same and hence the variation in the ratio of concentration of the components change the surface activity, which regulates the contact angle. We also plot the contact angle θ with the total concentration of the surfactant for some systems at room temperature ($\sim 30^\circ\text{C}$) (Fig. 2). A break point is observed. The values can be taken as CMC (see Table 1). However, we were not in a position to control the temperature of the teflon surface and the droplet very effectively. If that could be done then this would be an attractive procedure (non-intrusive) to obtain CMC, particularly of nonionic surfactants. However measurement of surface tension itself is an attractive non-intrusive procedure. Foaming of a surfactant solution is very important

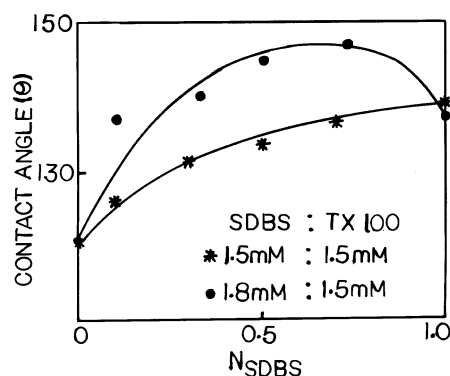


Fig. 1 Plots of contact angle vs mole fraction of SDBS. Various amounts of TX 100 (1.5 mM) solution was added to different amounts SDBS solution of 1.5 mM (*) and 1.8 mM (●), total volume 1 ml

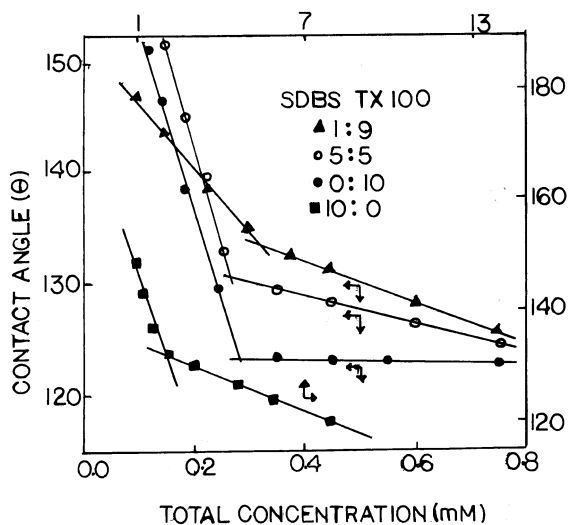


Fig. 2 Variation of contact angle with total concentration of surfactants at room temperature

property both in the consumer and non-consumer industries. In general anionic surfactants are known to be highly foaming and nonionic surfactants are non-foaming. A mixture of these two types of surfactants seems to show some what synergistic foaming property (Fig. 3). It can be observed from this figure that the foam height is a function of the concentration of the surfactants, the surfactant ratio and the temperature [28]. It is well known that the foams are very complex [41] and it seems from the figure that a clear relation between foam height and the variables does not exist. Moreover drainage, evaporation, interaction between environments and foam etc. also determine the foam stability [41]. The foam happens to occur be-

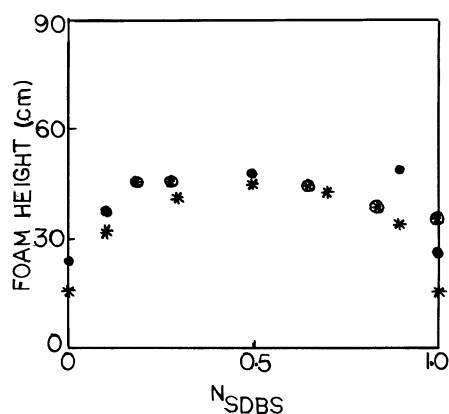
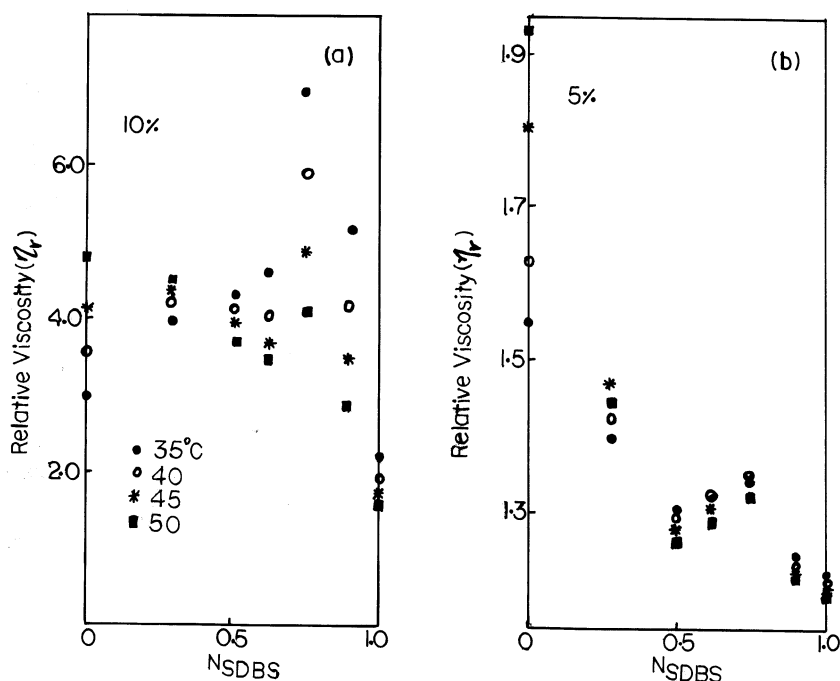


Fig. 3 Plots of foam heights vs mole fraction of SDBS. Various volumes of 0.238 M TX 100 solution was added to different volumes of * 0.238 mM; ○ 0.5 mM at 50 °C and ● 0.238 mM SDBS solutions at 35 °C. Total volume 300 ml

cause of the lower interfacial tension. Our studies were always below CMC. More importantly the foams are stable, if there is stability of the films at the air-water interface and also where surface tension varies rapidly with concentration [42]. We postulate that in the mixed systems, the possibility of rapid variation of concentration at the interface is higher than in the pure systems. Initial foam heights are reasonably stable (i.e. foams have maximum stability) just below the CMC [43]. Our foaming experiments were done much below CMC. The higher initial foam heights for mixed surfactant systems indicate increased interfacial activity. The plot (Fig. 3) indicates that the pure surfactants individually do have almost the same property but when concentration ratios are changed the foaming properties increase and seem somewhat independent of composition.

Viscosity is another important property that we studied. In Fig. 4a the relative viscosity for a 10% surfactant solution as a function of mole fraction of SDBS at various temperatures was plotted. The solution was Newtonian in nature at this concentration. It is obvious that at the mole fraction ~ 0.75 a maximum is observed at all temperatures indicating that around this composition the viscosity is maximum. The maximum in viscosity arises because of the formation of the mixed micelle as well as because of the electroviscous property. The electroviscous effect of mixed micelles is larger than that of single solution [44]. It should also be noted that at all temperatures the relative viscosity shows positive deviation from linearity indicating mixed micelle formation. One can also observe that as the concentration of SDBS increases, the relative viscosity at lower temperature increases more rapidly than at higher temperature because of electroviscous effect. However, the relative viscosity of a 5% solution (Fig. 4b) behaves somewhat differently from the 10% solution

Fig. 4 Plots of relative viscosity vs mole fraction of SDBS: (a) 10% mixtures; (b) 5% mixture



though, it shows a maximum at 0.75 mole fraction of SDBS. Another interesting feature is the fact that the viscosity of 5% surfactant solution in general show negative deviation from linearity. However, as is seen in the next para, the 5% solution has more spherical particles whereas the 10% solution has more non-spherical, i.e. anisometric micelle which probably effect the overall viscosity property.

The intrinsic viscosity $[\eta]$ can be calculated by the following relation:

$$[\eta] = \lim_{C \rightarrow 0} \frac{\eta_r - 1}{C},$$

where limit to zero concentration indicates that the inter-particle interactions are absent. The value of intrinsic viscosity is dependent on hydration of the surfactant particles as well as the size and shape. Some researchers have taken [45,46], $[\eta]$ to be equal to $(\eta_r - 1)/C$ without the condition of limiting concentration. It has been defined to be the “shape factor” [47] and is expected to have a value of $2.5\text{--}4\text{ cm}^3\text{ g}^{-1}$ for globular particles. If the limit of concentration is taken to zero then the effect of concentration on the geometry of the particle is lost and hence we computed the shape factor without taking limit to zero as was done by other researchers [45–47]. The maximum $[\eta]$ at 10% solution system was found to be $60\text{ cm}^3\text{ g}^{-1}$ at 35°C without taking zero concentration limit. This type of value indicates nonspherical particle rather than a spheri-

cal one [48]. Even in 5% solution, the lowest value is $\sim 5\text{ cm}^3\text{ g}^{-1}$ indicating that most of the particles are not spherical in nature [43]. It is known that the micellar structures become elongated and nonspherical as the concentration of the surfactant increases [49]. It is also accepted that Triton X 100 micelle is nonspherical [50]. The addition of SDBS should effect the micellar curvature to some extent. However, we do not expect the change to be so much as for the system to become spherical. Geometry of the cylindrical micelles is very dependent on the ratio of the two components [51]. The computed shape factor indicates the shape to be nonspherical in nature. A 5% (w/v) TX 100 solution is ~ 350 times more concentrated than the CMC of TX 100. It can be noted from Fig. 4a and b that for TX 100, the η_r increases with increase in temperature. This is expected as TX 100 shows cloud point. For SDBS and most of the SDBS–TX 100 systems (10%), the expected decrease in η_r with increase in temperature was observed. Eyring equation

$$\eta = \eta_0 \exp(\Delta G_{\text{vis}}^{\ddagger}/RT),$$

where η and η_0 are the viscosities of solution and solvent respectively and other symbols have their usual meanings, was used to obtain free energy of activation of the flow process $\Delta G_{\text{vis}}^{\ddagger}$. This quantity measures the nonmechanical amount of energy that must be spent for the flow process to occur. From Fig. 5 it is quite clear that the variation of $\Delta G_{\text{vis}}^{\ddagger}$ with mole fraction of SDBS shows a maximum. This variation is also a function of total concentration. At lower

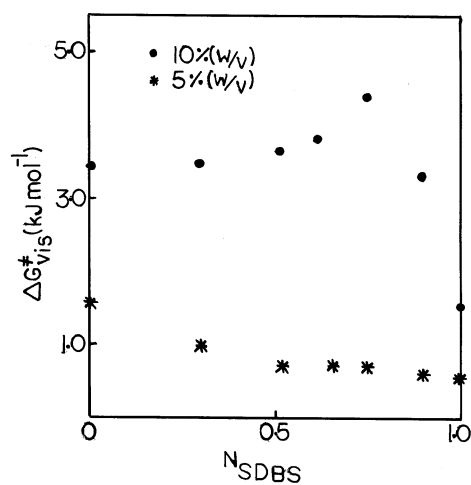


Fig. 5 Plots of $\Delta G_{vis}^{\ddagger}$ vs mole fraction of SDBS. * 5% and ● 10% mixture

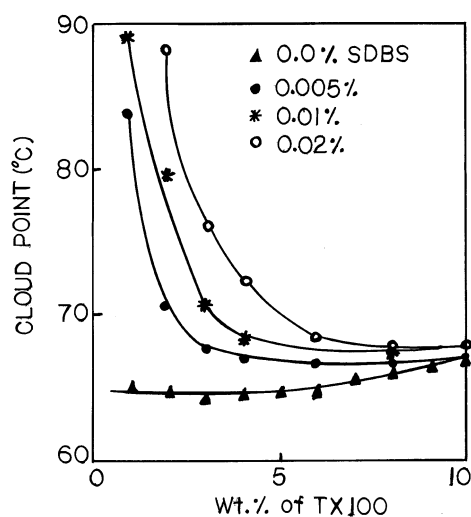


Fig. 6 Plots of cloud points vs concentration of TX 100 in the presence of SDBS

total concentration of 5% it seems that $\Delta G_{vis}^{\ddagger}$ becomes very low and constant when mole fraction of SDBS was over 0.5. In other words the nature of the solution becomes independent of mixture composition.

In Fig. 6 cloud point of various Triton X 100 solutions are shown in presence of different concentrations of SDBS. As the concentration of TX 100 increases the cloud point decreases. However, as was shown earlier [52] the cloud points show a minimum at ~3%. In the present case where ionic SDBS is present, such minimum was not observed. Cloud point continues to decrease and becomes reasonably constant at higher Triton concentration. Generally at higher concentration i.e. over 3% the cloud point

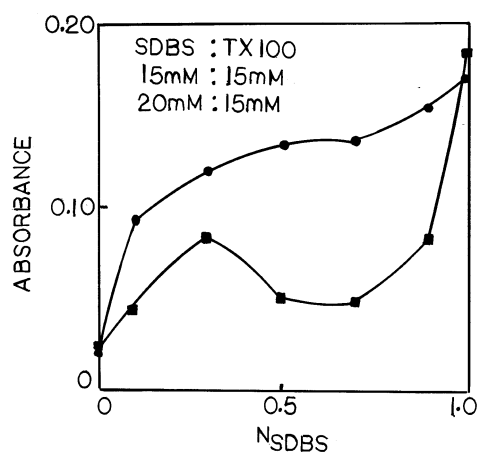


Fig. 7 Plots of absorbance vs mole fraction of SDBS at 10 min. To a fixed volume of 15 mM TX 100 solution various volumes of 15 mM SDBS (■) and 20 mM SDBS solution were added (●)

increases because of the presence of water molecules as a part of the total system [53]. Higher temperature is required to remove this water and hence higher cloud point. However in presence of SDBS this water cannot be present because of the solvation of Na^+ . Hence higher temperature is not needed. The cloud point decreases smoothly to become more or less constant. The cloud point at a definite concentration of Triton decreases with higher SDBS concentration. Because SDBS forms a mixed micelle, the micelle is charged. Therefore higher temperature is needed for the micelle to come together overcoming their coulombic repulsion.

We also studied the removal of dye Malachite green from cloth. From Fig. 7 it can be concluded that the removal of the dye is favoured by the mixed system rather than by pure TX 100. The pure SDBS solution, of course, is more efficient. An expected feature of the graph is the fact that at higher amount of mixed system the dye removal efficiency was higher. However, it is interesting to note that there is deep around 0.7 mole fraction of SDBS in both cases. From Fig. 4a and b it was noted that the relative viscosity shows maximum around this mole fraction ratio. Though absolute concentrations used in both cases (i.e. Fig. 4a, b and Fig. 7) are very different, we can say that least efficiency around 0.75 mole fraction SDBS concentration probably arises due to relatively higher viscosity of this mixed system. In conclusion it can be said that the properties of mixed SDBS–TX 100 solutions are quite understandable though a bit difficult to predict. The mixed micelle formed are ionic and Rubingh's interaction parameter is not always possible to compute. The wetting characteristic seems to be useful to determine CMC as a nonintrusive method as the measurement of surface tension is.

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