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ORIGINAL ARTICLE

Micellization Behavior of Mixtures of Sodium Dioctylsulfosuccinate with Sodium Dodecylsulfate in Water

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Abstract Mixtures of sodium dioctylsulfosuccinate (AOT) and sodium dodecylsulfate (SDS) that were studied in water at 25 °C by using surface tension, conductance, emf and fluorescence emission methods exhibit synergism in the region where the mole fraction of AOT in the bulk solution (α_1) is less than 0.7 and ideality in the region where $\alpha_1 \geq 0.7$. The molal conductance versus the concentration behavior of an aqueous solution of AOT is found to be different from that of other ionic surfactants with the exception of bile salts. Composition of the mixed micelle was evaluated and discussed using the Rubingh's and the Rodenas–Valiente–Villafruela (RVV) treatments. The values of the counter ion binding constant determined from the emf data show that the counter ion binding behavior of the mixed micelle is controlled entirely by AOT. The free energy for mixed micelle formation was calculated using a modified equation. The aggregation number determined by the fluorescence quenching method indicated that in the mixed micelle, as α_1 increases, the number of molecules of AOT remains constant and that of SDS decreases. Characteristics of the adsorption layer of the mixed surfactant system were also examined using the theoretical treatment of Rosen and Hua.

Keywords SDS · AOT · Mixed micelle · Critical micelle concentration · Counter ion binding constant · Surface excess · Free energy · Aggregation number

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Introduction

Compared to single surfactants, mixtures of surfactants perform better and hence formulations developed for different applications in various fields consist of mixed surfactants. Investigation of the micellization properties of mixed surfactants has therefore become fundamental as well as of industrial importance. For forming mixtures of surfactants different combinations of single surfactants are possible. Mixtures of dissimilar types of surfactant ordinarily exhibit considerable synergism. Mixtures of similar type of surfactants are also known to exhibit synergism and among them combinations of same type of ionic surfactants have been studied relatively less frequently than the mixtures of nonionic–nonionic surfactants [1–9]. In particular, mixed systems containing double-chained and single-chained ionic surfactants have not been explored much [3,7–9]. Sodium dioctylsulfosuccinate (AOT) is a widely used double-chained anionic surfactant, because it forms microemulsions without any cosurfactant. To the best of our knowledge, no report is available on the behavior of mixtures of AOT with other anionic surfactants, although one report was found on mixtures of AOT with a cationic surfactant, cetyltrimethylammonium bromide [10]. Therefore, in the present article the micellization properties of mixtures of AOT and sodium dodecylsulfate (SDS) in water have been studied at 25 °C by using surface tension, emf, conductance and fluorescence emission methods.

Experimental Procedures

AOT (Sigma, 99% assay), SDS (Aldrich, 99% assay), pyrene (Fluka, 99% assay) and cetylpyridinium chloride (CPC; Fluka, 98% assay) were used as received. All

solutions were prepared in Millipore water and measurements were made under thermostated condition at 25 °C. An aqueous solution containing a mixture of AOT (surfactant 1) and SDS (surfactant 2) of a particular composition was prepared by mixing required amounts of the solutions of pure surfactants.

Surface tension measurements were made by the Wilhelmy plate method using a Krüss K11 tensiometer. Conductance measurements were made using a Wayne Kerr B905 Automatic Precision Bridge. EMF measurements were made using a Jenway 3345 model Ionmeter and a Jenway 924-329 combined ion-selective electrode reversible to sodium ion concentration in the solution. The fluorescence emission measurements of the pyrene probe were made using a Hitachi F4500 model Fluorescence Spectrophotometer. The excitation of pyrene was done at 335 nm and the fluorescence emission was recorded in the range of 360–420 nm. CPC was used for fluorescence quenching.

Results and Discussion

Representative plots of measured surface tension (γ) versus surfactant concentration are shown in Fig. 1. The SDS sample used in this study exhibited a surface tension minimum and the depth of the minimum decreased with the addition of AOT. Occurrence of such a surface tension minimum for SDS and its disappearance on adding more sodium ions have been discussed in a recent paper [11]. Representative plots of measured emf as a function of surfactant concentration are presented in Fig. 2. A few representative plots of I_3/I_1 ratio versus surfactant concentration are shown in Fig. 3. I_1 and I_3 denote the intensities of fluorescence emission of pyrene corresponding to 373 and 384 nm, respectively.

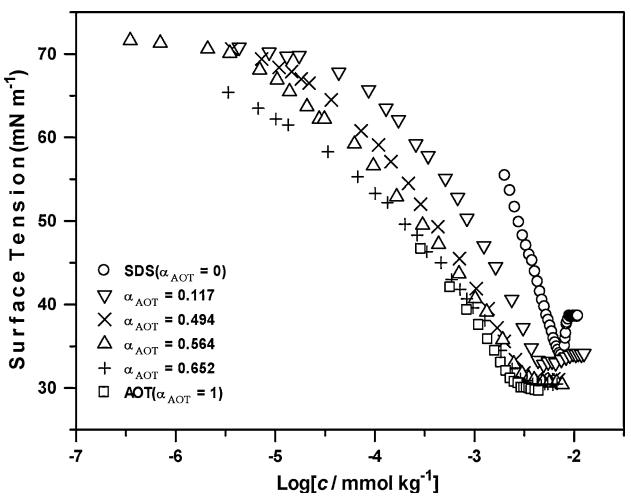

Fig. 1 Representative plots of surface tension of AOT + SDS system against the logarithm of the total surfactant concentration

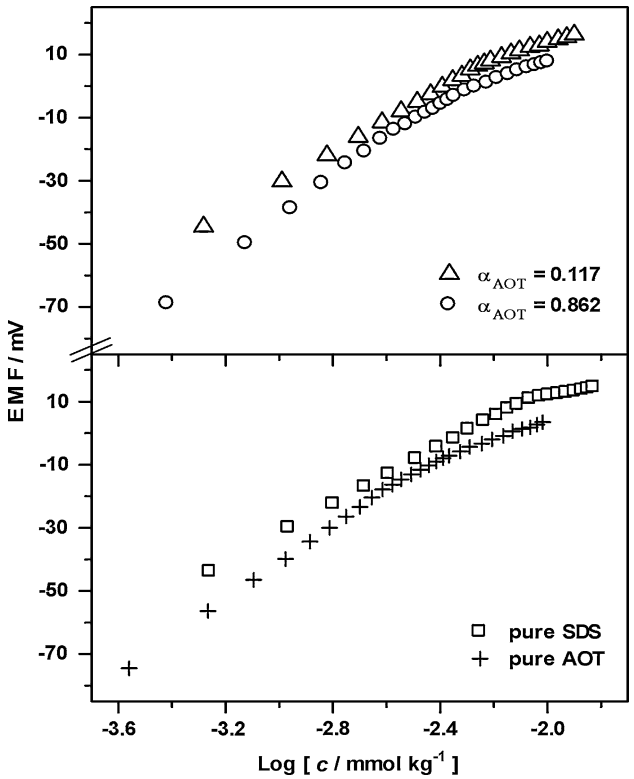

Fig. 2 Representative plots of emf of AOT + SDS system against the logarithm of the total surfactant concentration

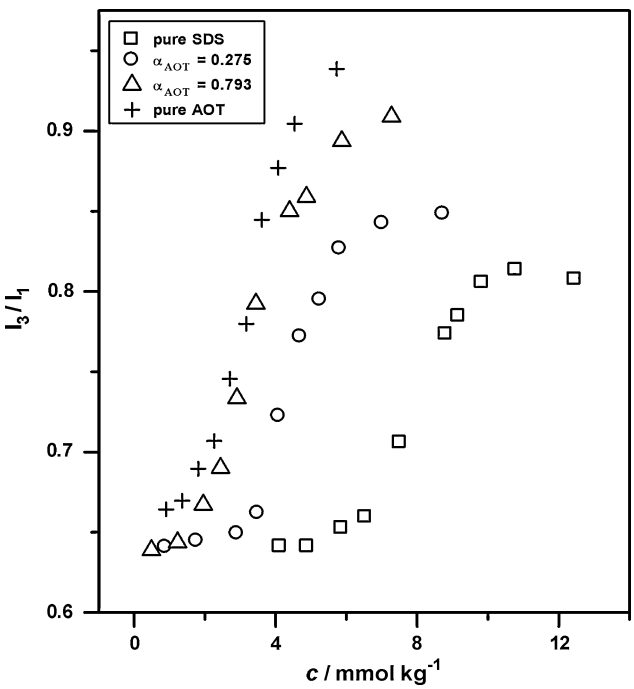

Fig. 3 Representative plots of I_3/I_1 values of pyrene in AOT + SDS system against the total surfactant concentration

It has generally been considered that the cmc of AOT cannot be determined correctly from the plot of specific conductance versus concentration [12] unlike the case with

other ionic surfactants. The molal conductance (Λ) of the surfactant solutions was therefore calculated and a few representative plots of Λ versus the square root of the concentration are shown in Fig. 4. Surprisingly, such a plot for AOT exhibits a minimum followed by a maximum and the concentration at the minimum is found to correspond to the cmc of AOT. Thus, it is possible to determine accurately the cmc of AOT from the molal conductance data rather than from specific conductance data. By the addition of SDS to AOT, the Λ minimum gradually disappears and no minimum occurs in the region where the mole fraction of AOT ≤ 0.8 (Fig. 4). This type of variation of Λ with concentration is generally not observed for ionic surfactants. Bile salts are the only other example where a similar trend has been reported [13–15]. Although in bile salts occurrence of conductance minimum and maximum is ascribed to the presence of a dynamical equilibrium between transient structures like dimers and trimers [13–15], the species responsible for the type of molal conductance behavior shown by AOT are not known at the moment.

The Cmc of Mixed Surfactants

The values of the cmc of the mixed surfactants (c_{0m}) determined by the various methods are shown in Fig. 5 as a plot of c_{0m} versus mole fraction of AOT in the bulk solution (α_1). Relationship between c_{0m} and the cmc's of AOT (c_{01}) and SDS (c_{02}) is of the form [16, 17]

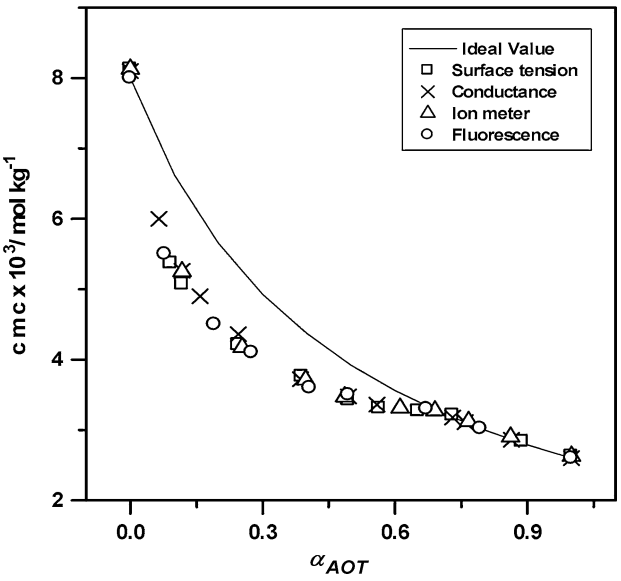

Fig. 5 Values of cmc of AOT + SDS system obtained from surface tension, conductance, emf and fluorescence methods as a function of the mole fraction of AOT in the bulk solution. The ideal value of cmc is calculated from Clint's equation

$$1/c_{0m} = \sum_i [\alpha_i/(f_i c_{0i})] \quad (1)$$

$$f_i = \alpha_i c_{0m}/(x_i c_{0i}) \quad (2)$$

In Eqs. (1) and (2) x_i and f_i refer to the mole fraction and activity coefficient of AOT ($i = 1$) or SDS ($i = 2$) in the mixed micelle, respectively. When the mixed micelle is assumed to behave ideally ($f_i = 1$), Eq. (1) reduces to the Clint's equation [18]. From Fig. 5, it is apparent that the

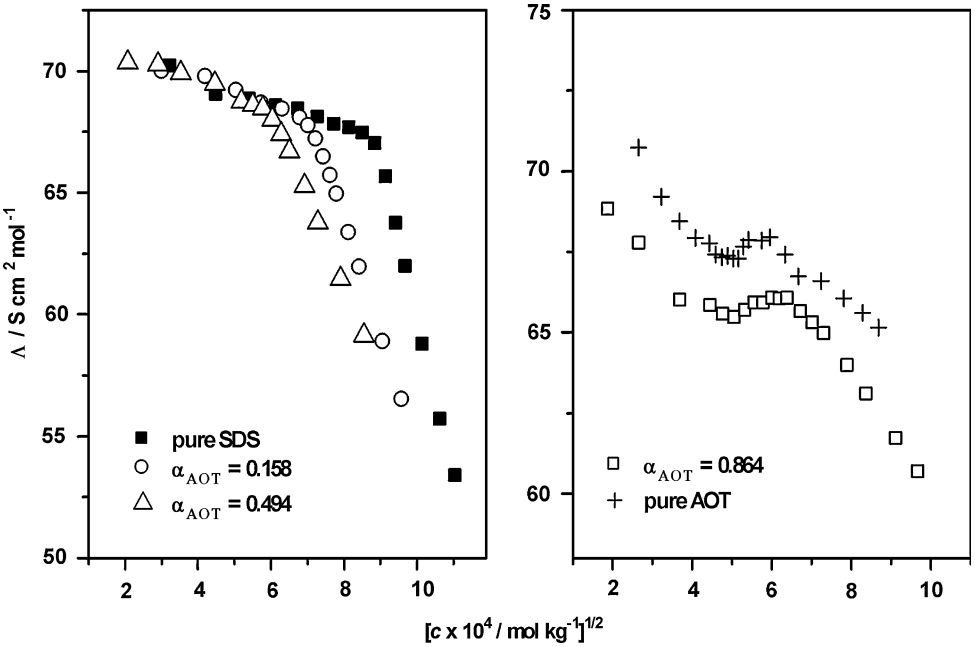

Fig. 4 Plots of the molal conductance of the AOT + SDS system versus the square root of the total surfactant concentration

experimental values of c_{0m} are less than the ideal values thereby indicating synergism for compositions less than the 0.7 mole fraction of AOT. In the region of $0.7 \leq \alpha_1 \leq 1$, c_{0m} takes up ideal values.

The Mixed Micelle Composition by Rubingh's Method

In this method [16, 17], the regular solution approximation is employed to describe the non-ideality of the mixed micelle. According to this approximation, the relation between the interaction parameter (β_m) responsible for non-ideality and the activity coefficient is given by

$$\beta_m = \ln f_1/(1-x_1)^2 = \ln f_2/x_1^2 \quad (3)$$

At c_{0m}, after substituting for the activity coefficients in Eq. (3), we get the expression

$$x_1^2 \ln[\alpha_1 c_{0m}/(x_1 c_{01})] = (1-x_1)^2 \ln[(1-\alpha_1)c_{0m}/\{(1-x_1)c_{02}\}] \quad (4)$$

Values of x_1 as a function of α_1 were computed from Eq. (4) by using an iterative method and are shown in Fig. 6 as x_1^{Rb}. It is evident from Eq. (2) that the value of x_i is controlled by the ratio c_{0m}/c_{0i} and therefore $x_1 > \alpha_1$ and $x_2 < \alpha_2$. An expression for x_1 was also derived from Eq. (2) under the condition of ideality and it is of the form

$$x_1 = \alpha_1 c_{02}/(\alpha_1 c_{02} + \alpha_1 c_{01}) \quad (5)$$

The values of x_1 calculated from Eq. (5) are also shown in Fig. 6 as x_1^{id}. From Fig. 6, it may be observed that (1) $x_1^{Rb} > x_1^{id}$ in the region where $\alpha_1 < 0.25$, (2) $x_1^{Rb} < x_1^{id}$ in the region where $0.38 < \alpha_1 < 0.73$ and (3) $x_1^{Rb} = x_1^{id}$ in the region where $\alpha_1 \geq 0.73$. To explain this type of trend in the values of x_1^{Rb} and x_1^{id}, the values of f_i were first calculated from Eq. (2) after substituting the values of x_1^{Rb} for x_1. The values of f_i calculated in this way are presented in Fig. 6. Equation (2) can now be written as $x_1^{Rb} = x_1^{id}/f_1$, which reveals that $x_1^{Rb} > x_1^{id}$ since $f_1 < 1$. This condition must be applicable in the region where $\alpha_1 < 0.25$. On the other hand, Eq. (2) can also be written as $x_1^{Rb} = 1 - \alpha_2 c_{0m}/(f_2 c_{02})$ and $x_1^{id} = 1 - \alpha_2 c_{0m}/c_{02}$, which reveals that $x_1^{Rb} < x_1^{id}$ since $f_2 < 1$. This condition must be applicable in the region where $0.38 < \alpha_1 < 0.73$. In the region where $\alpha_1 \geq 0.73$, $f_1 = f_2 = 1$ and hence $x_1^{Rb} = x_1^{id}$. Thus, the value of x_1^{Rb} is controlled by f_1 in the region where $\alpha_1 < 0.25$ and by f_2 in the region where $0.38 < \alpha_1 < 0.73$.

The values of the interaction parameter β_m were determined using Eq. (3) and are presented in Fig. 7. Negative values of β_m are indicative of the attractive interaction between the two anionic surfactants. The magnitude of β_m becomes less negative with increasing mole fraction of AOT and finally reaches zero above the 0.7 mole fraction of AOT (Fig. 7). An initial addition of a small amount of AOT to SDS causes a sharp change in the interaction parameter and further addition of AOT weakens the interaction (Fig. 7).

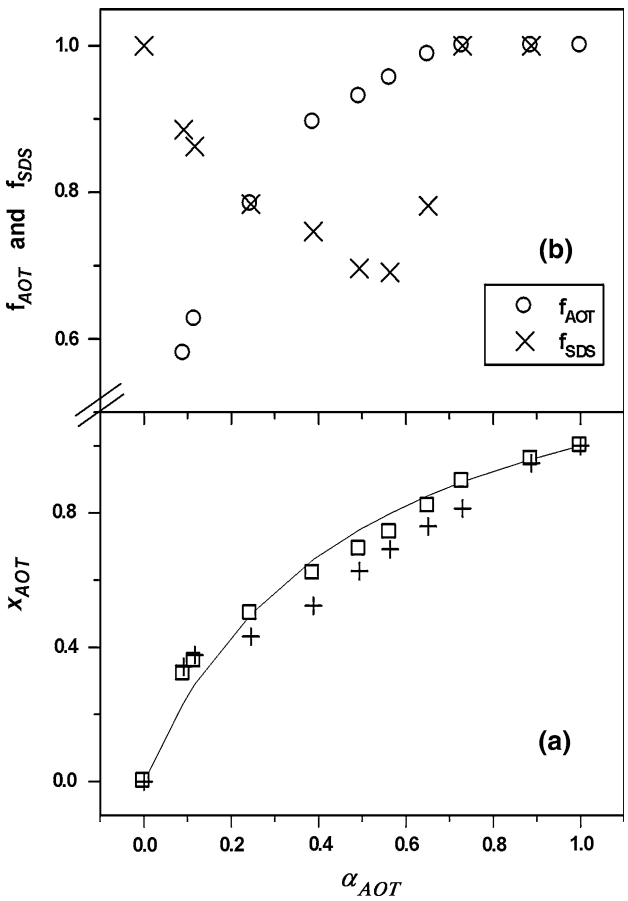

Fig. 6 Plots of **a** the mole fraction of AOT (x_{AOT}) and **b** activity coefficients of AOT (f_{AOT}) and SDS (f_{SDS}) in the mixed micelle against the mole fraction of AOT in the bulk solution. Values of x_{AOT} were calculated from (1) Rubingh's method [Eq. (4), x_{AOT}^{Rb} (*open squares*)], (2) RVV method [Eq. (6), x_{AOT}^{Rd} (+)] and (3) by considering ideal condition [Eq. (5), x_{AOT}^{id} (–)]

It is worthwhile comparing the micellization behavior of the AOT + SDS mixed system with that of other anionic–anionic mixed surfactant systems having SDS as one of the components and sodium as the only counterion. In the case of STSa (sodium tetradecylsarcosinate) + SDS system [19] the mixing was reported to be ideal. On the other hand, SDBS (sodium dodecyl benzene sulfonate) + SDS system was reported [19] to show marginal antagonistic behavior with the cmc of the mixture having values slightly higher (maximum deviation = 3.5%) than the ideal values (average $\beta_m = 0.66$). In the sodium tetradecylsulfate (STS) + SDS system [2], synergism was found with the cmc of the mixture having a maximum deviation of 8.5% from the ideal value (average $\beta_m = -0.40$). In the AOT + SDS system, as mentioned above, synergism is shown only in the region where $\alpha_1 < 0.7$ with maximum deviation of 22.2% from the ideal value.

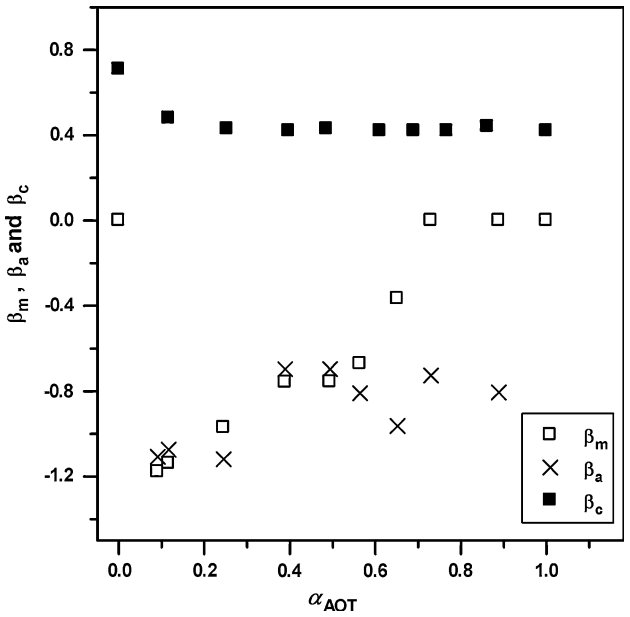

Fig. 7 Values of the interaction parameter in the mixed micelle (β_m), the interaction parameter in the adsorbed layer (β_a) and the counter ion binding constant (β_c) as a function of the mole fraction of AOT in the bulk solution

The Mixed Micelle Composition by the RVV Method

Rodenas et al. [20] proposed an alternative approach to determine x_1 by using the Gibbs-Duhem relation and without resorting to the regular solution approximation. According to this method, the equation for x_1 is of the form

$$x_1 = \alpha_1 - \alpha_1(1-\alpha_1)d\ln c_{0m}/d\alpha_1 \quad (6)$$

For computing the values of x_1 from Eq. (6), the value of the slope, $d \ln c_{0m}/d\alpha_1$, was determined first by least-squares fitting the c_{0m} versus α_1 data to an empirical relation of the form

$$\ln c_{0m} = \sum_j a_j \alpha_1^j, \quad j = 0 \text{ to } 5 \quad (7)$$

The least-squares fitted values of the empirical coefficients of Eq. (7) are $a_0 = -4.820$, $a_1 = -5.732$, $a_2 = 18.859$, $a_3 = -33.805$, $a_4 = 29.782$ and $a_5 = -10.231$. The values of x_1 calculated from Eq. (6) are presented in Fig. 6 as x_1^{Rd}. The deviation of x_1^{Rd} from x_1^{id} is found to be more than that of x_1^{Rb} (Fig. 6).

The Dependence of the Mixed Micelle Composition on the Concentration of the Mixed Surfactants

It may be noted that the values of x_1 evaluated by Rubingh's and RVV methods are valid only at c_{0m}. The composition of the mixed micelle may be expected to vary with the total surfactant concentration (c). To obtain information about the dependence of x_1 on c, x_1 is written on the basis of its definition as [20]

$$x_1 = (\alpha_1 c - c_1)/(c - c_1 - c_2) \quad (8)$$

In the region where $c \geq c_{0m}$ if we presume that the concentrations of the surfactants 1 (c_1) and 2 (c_2) in the bulk solution remain constant and are given by $c_i = \alpha_i c_{0m}$, then Eq. (8) reduces to the form $x_1 = \alpha_1$. However, since x_1 is found to be greater than α_1 (Fig. 6), it is implied that c_1 and c_2 do not remain constant above the cmc and vary with c. Since above the cmc, equilibrium also exists between a surfactant in the free and mixed micellar forms, we get from the expressions for the chemical potential terms that $c_1 = x_1\ f_1\ c_{01}$ and $c_2 = x_2\ f_2\ c_{02}$. By substituting these values for c_1 and c_2 in Eq. (8), we obtain a quadratic equation [20] of the form

$$x_1^2(c_{01}f_1 - c_{02}f_2) - x_1(c + c_{01}f_1 - c_{02}f_2) + \alpha_1 c = 0 \quad (9)$$

Equation (9) was solved for different values of c after substituting the values of f_1 and f_2. While solving Eq. (9) for a chosen value of α_1, f_1 and f_2 were presumed to be independent of c. The values of x_1 so obtained from Eq. (9) are shown as a function of c in Fig. 8. From Fig. 8 it is apparent that x_1 approaches the value of α_1 at $c \geq 0.1$ mol kg^{-1}. This can be explained on the basis of Eq. (9) itself because on rearrangement it takes the form

$$x_1 x_2(c_{02}f_2 - c_{01}f_1) + \alpha_1 c - x_1 c = 0 \quad (10)$$

At high values of c, in the present case above 0.1 mol kg^{-1}, the first term of Eq. (10) becomes negligible making $x_1 = \alpha_1$.

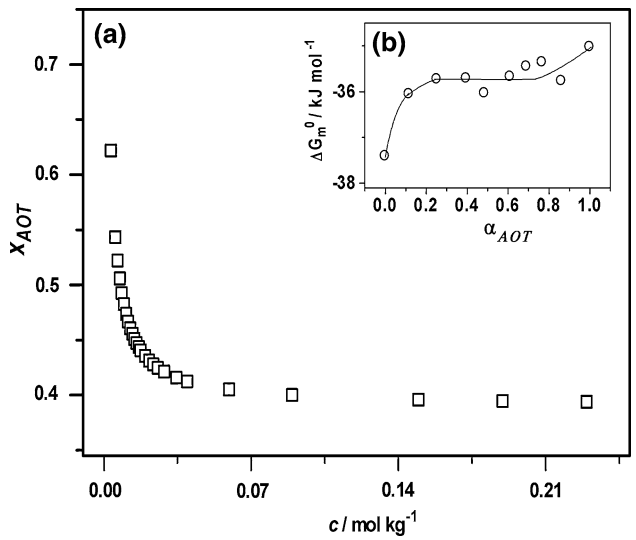

Fig. 8 Plots of **a** the mole fraction of AOT in the mixed micelle against the total surfactant concentration above the cmc and corresponding to $\alpha_{AOT} = 0.389$ and **b** standard free energy for mixed micelle formation against the mole fraction of AOT in the bulk solution

Counter Ion Binding to the Mixed Micelle

From the emf (E) data, the counter ion binding constant β_c was determined as described elsewhere [21] and the calculated values of β_c are presented in Fig. 7. In the mixed micelle, it is interesting to note that β_c has a value that is characteristic of AOT [21] even if the proportion of SDS is more in the mixed micelle (Fig. 7). This envisages that counter ion binding of the mixed micelle is controlled entirely by the AOT thereby indicating that, in the Stern layer of the mixed micelle, heads of AOT are probably more exposed than those of SDS.

Free Energy of Mixed Micelle Formation

For calculating the free energy of the mixed micelle formation the same expression as that applicable in the case of the single surfactant solution is commonly used [5,22,23]. The expressions for the free energy of micelle formation in the cases of mixed and pure surfactants can be different. To show this, the following equilibrium is considered for a mixture of two anionic surfactants containing the same counter ion (R^+):

$$n_1 S_1^- + n_2 S_2^- + mR^+ \rightleftharpoons M^{(n_1+n_2-m)-} \tag{11}$$

In this equilibrium, M refers to the mixed ionic micelle and m is the number of counter ions that binds to a mixed micelle. n_1 and n_2 indicate the number of molecules of surfactants RS_1 and RS_2, respectively that aggregate to form the mixed micelle. In the present system, the charges of S_1, S_2 and R are equal to one. Based on thermodynamics, an expression for the standard free energy change (ΔG°) for the above equilibrium process can be written and rearranged to the form

$$x_1 \ln[S_1^-] + x_2 \ln[S_2^-] \approx [\Delta G^\circ/(N_o RT)] - \beta_c \ln[R^+] \tag{12}$$

where $N_o = n_1 + n_2$ is the aggregation number of the mixed surfactant system and $x_1 = n_1/(n_1 + n_2)$. In writing Eq. (12), the value of $\ln[M]$ has been neglected in comparison to $\Delta G^\circ/(RT)$, which is an approximation made in the case of single surfactant solutions as well. At the cmc of the mixed surfactant solution of a particular composition (α_1) and in the absence of added electrolyte, Eq. (12) can be written as

$$\Delta G_m^\circ = RT[(1+\beta_c)\ln c_{0m} + x_1 \ln \alpha_1 + x_2 \ln \alpha_2] \tag{13}$$

where $\Delta G^\circ{}_m$ is the standard free energy of micellization of the mixed surfactants per monomer and is defined as $\Delta G^\circ{}_m = \Delta G^\circ/N_o$. Values of $\Delta G^\circ{}_m$ calculated by using Eq. (13) are presented in Fig. 8. In the composition region where $0.1 \lessapprox \alpha_1 \gtrapprox 0.7$ $\Delta G^\circ{}_m$ is found to be almost constant, which implies that, in this region, β_c controls the value of $\Delta G^\circ{}_m$ since β_c is constant in this region.

Adsorption Parameters

Using the Gibbs adsorption isotherm [24], it can be shown for a mixture of 1:1 ionic surfactants having same the counter ion that

$$d\gamma = -2RT\Gamma(d\ln c + x_{1a} d\ln \alpha_1 + x_{2a} d\ln \alpha_2) \tag{14}$$

where Γ is the surface excess of the mixed surfactant system. $x_{ia} = \Gamma_i/\Gamma$ is the mole fraction of the surfactant 1 ($i = 1$) or 2 ($i = 2$) in the adsorbed layer. In the case of a mixed ionic surfactant system having a particular composition (constant α_1), Eq. (14) becomes $d\gamma = -2RT\Gamma d\ln c$, which is the same equation used for calculating the surface

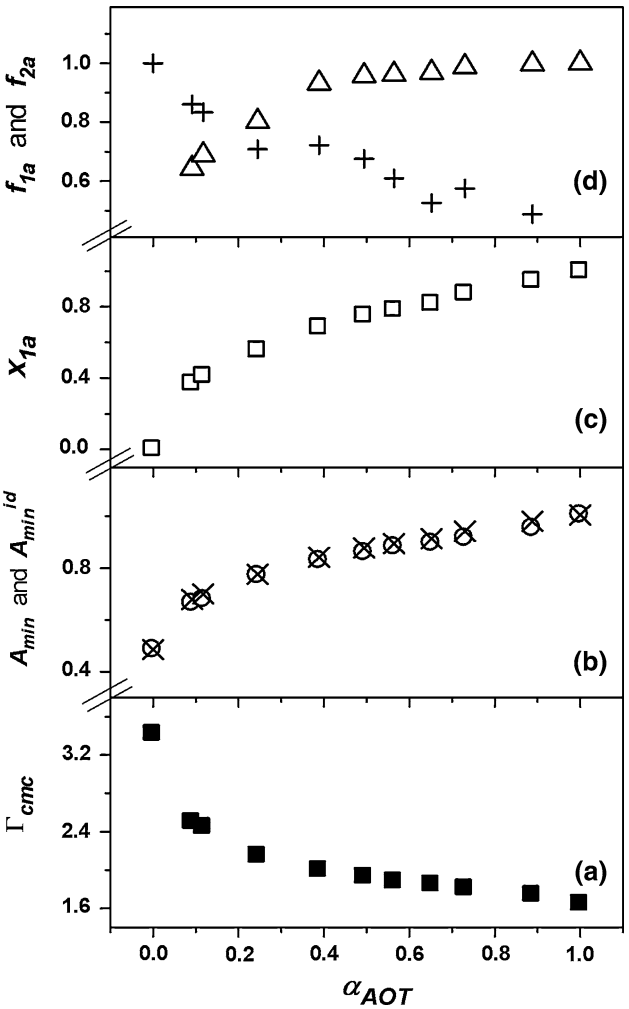

Fig. 9 Plots of **a** surface excess (10^{-6} mol m^{-2}) at the cmc, Γ_{cmc}, **b** area (nm^2) per molecule in the adsorbed layer, A_{min} (*open circles*) and A^{id}_{min} (×), **c** mole fraction of AOT in the adsorbed layer (x_{1a}), and **d** activity coefficients of AOT [f_{1a}, Δ] and SDS [f_{2a}, +] versus mole fraction of AOT in the bulk solution

excess of a single surfactant [24]. Accordingly, surface excess at the cmc (Γ_{cmc}) and the minimum surface area per mole of the surfactant mixture (A_{min}) were calculated [24] and are shown in Fig. 9. By applying Rubingh's approach to adsorption at a reference point of constant surface tension, Rosen and Hua [25] reported expressions for activity coefficient (f_{ia}), interaction parameter (β_a) and mole fraction (x_{ia}) of a surfactant in the adsorbed layer of mixed surfactants. Using those reported [25] expression values of f_{1a}, f_{2a} and x_{1a} were calculated and are shown in Fig. 9, while the calculated values of β_a are shown in Fig. 7. The ideal values of A_{min} calculated from the expression $A^{id}{}_{min} = x_{1a} A_{1\ min} + x_{2a} A_{2\ min}$ are also presented in Fig. 9. A_{1min} and $A_{2\ min}$ represent the A_{min} values for surfactants 1 and 2, respectively. The values of A_{min} are found to be almost equal to those of $A^{id}{}_{min}$. The values of β_a are negative (Fig. 7), which indicate an attractive interaction between the surfactant molecules adsorbed at the interface. The mole fraction of AOT at the adsorbed layer (x_{1a}) is more than the bulk mole fraction (α_1) indicating that the mixed adsorption layer is enriched with AOT and it is due to higher surface activity of AOT.

Aggregation Number

The aggregation number (N_o) of the mixed surfactant system was determined by measuring the fluorescence emission of pyrene in the absence and presence of the quencher CPC and using the reported [26] expressions. The measured values of N_o are presented in Fig. 10. The number of molecules of AOT (n_{AOT}) and SDS (n_{SDS}) in the mixed micelle were estimated from the values of N_o by using the mole fraction values of the individual surfactants in the mixed micelle. N_o increases by the initial addition of AOT to SDS and has a maximum at $\alpha_1 \approx 0.12$ (Fig. 10), while n_{AOT} is found to be almost constant (25 ± 5). Therefore, in the mixed micelle, the presence of SDS does not affect n_{AOT}, while n_{SDS} decreases by adding AOT (Fig. 10). Since we measured N_o of the mixed micelle by keeping the value of $c - c_{0m}$ constant at 0.005 ± 0.001 mol kg^{-1}, the observed trends in the variation of n_{AOT} and n_{SDS} with α_1 seem to indicate that n_{AOT} is influenced only by the amount of the counter ion, whereas n_{SDS} is dependent on the concentrations of both the counter ion and the dioctylsulfosuccinate ion. It may, however, be commented that constancy of n_{AOT} in the present mixed micelle may be a coincidence and to explain such a behavior further investigation of similar types of mixed micelles is required.

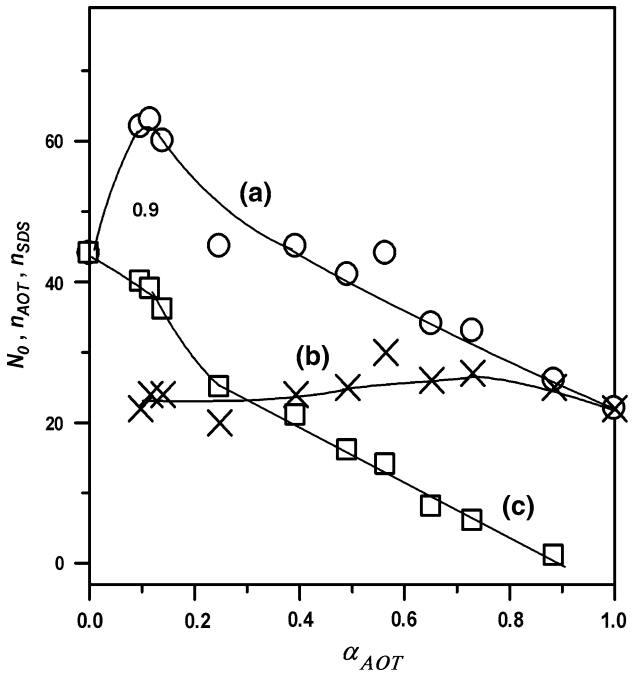

Fig. 10 Plots of **a** the aggregation number (N_o, *open circles*) of the AOT + SDS mixed system, **b** the number of molecules of AOT in the mixed micelle (n_{AOT}, ×), and **c** the number of molecules of SDS in the mixed micelle (n_{SDS}, *open squares*) against the mole fraction of AOT in the bulk solution

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