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ORIGINAL CONTRIBUTION

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Synergistic behavior of sodiumdodecylsulfate and 1,2-diheptanoyl-sn- glycero-3-phosphocholine in an aqueous medium: interfacial and bulk behavior

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Abstract The synergistic behavior of sodiumdodecylsulfate (SDS) and 1,2-diheptanoyl-sn-glycero-3-phosphocholine (DHPC) binary mixtures has been studied with interfacial and pyrene fluorescence(I_1/I_3) intensity measurements. From the interfacial data, the interfacial parameters; the maximum surface excess (Γ_{max}), or the minimum area per molecule (A_{min}), and the surface pressure at the critical micellar concentration (π_{cmc}) have been evaluated. The cmc value has been used for evaluating the free energies of micellization ($\Delta G^{o}{}_{m}$). The mixed micelle formation was evaluated with the help of the Clint equation. The SDS plus DHPC mixed micelles showed negative departure from ideality indicating synergistic interactions between the unlike components. The quantitative analysis of mixed micelle, mixed monolayer and the composition of the mixed micelle was carried out with the help of regular solution approximation. The interaction parameters, β and β^{σ}, in the mixed micelle as well as in the mixed monolayer, respectively showed negative values indicating synergistic behavior of SDS and DHPC molecules.

Keywords SDS · DHPC · Mixed micelles · Interfacial · Fluorescence behaviors

Introduction

Surfactants are surface active agents consisting of a hydrophilic and hydrophobic moiety. This dual nature gives rise to some remarkable properties. When a surfactant is dissolved in water, the hydrophobic moiety forms cavities within the hydrogen-bonding network of water molecules distorting the water structure. The distortion decreases the entropy of the system, resulting in an increase in free energy. To minimize contact with the water molecules, the individual surfactant molecules or binary mixtures form a variety of aggregates in the bulk of the solution at a certain concentration known as the critical micelle concentration (cmc). The structure of these aggregates significantly depends upon their concentration, presence of salt, and temperature [1–5].

On the other hand, phospholipids are naturally occurring surface active compounds with a polar head group and a nonpolar hydrophobic tail. Phospholipid molecules with short hydrocarbon chains form spherical, disklike, or cylindrical micelles in water, just like the conventional amphiphiles [1, 2]. During the last decade, the properties of conventional mixed surfactants have been studied intensively with regard to the behavior of mixed monolayer and micelle formation [6–15]. The binary mixtures of surfactant and phospholipids have been somewhat neglected, however. Mixed micelle formation has considerable importance in comparison to single surfactant micelle formation ranging from various applied aspects such as detergency, wetting, spreading, foaming etc especially when one component consists of a double hydrocarbon tail of lipid [1, 2, 5]. The mixed system almost invariably yields enhanced interfacial properties (e.g. decreased cmc and higher surface activity) in comparison to individual surfactants, a phenomenon known as synergism.

In different combinations of nonionic, ionic and zwitterionic surfactants, it is known that nonionic mixtures show ideal behaviors [6, 7], while the other pairs usually show nonideality resulting from the synergistic (attractive) or antagonistic (repulsive) interactions between the surfactant's monomers [8–15]. Theories developed by Clint [16], Motumura [17], Rubingh [18] and Blackschtein [19] have been extensively used to analyze and compare the experimental results to reveal the synergistic and antagonistic behaviors of different binary combinations. Binary mixtures exhibiting synergism have many useful applications, but the molecular processes are relatively poorly understood. This makes it particularly relevant to study the synergistic behavior of surfactant and short chain phospholipids (lecithins) due to their dissimilar hydrophobicities and head group polarities.

The present article deals with the properties of the monolayer at the air-water interface and the micellar behavior of binary mixtures of sodiumdodecylsulfate (SDS) and phospholipid 1,2-diheptanoyl-sn-glycero-3-phosphocholine (DHPC) in order to evaluate their synergistic interactions due to their significant differences in molecular structure. The theories of Clint and regular solution approximation have been used to evaluate the cmc, composition and interaction parameters of the two components. The DHPC containing two C-7 chains was chosen because of its ability to form micelles in water, whereas phospholipids, with chain lengths of C-12 and above, exist mainly as bilayer assemblies [1, 2, 5].

Experimental

Materials

Phospholipid DHPC (≥ 99% pure) was obtained from Avanti Polar Lipids, Inc., USA and used as received. SDS from Fluka, Buchs, Switzerland, (≥ 99% pure) was used as received. Pyrene was purchased from Nacalai Tesque, Kyoto, Japan. All solutions were prepared in Millie-Q water having specific conductance of 2 μS cm^{-1}.

Methods

The interfacial tension was measured with the help of Interfacial Tensiometer Model PD-X (pendant drop method) from Kyowa Interface Science Co. Ltd., Saitama, Japan. A drop of sample was formed by using a 1 cm^3 syringe, fitted with a 22 gauge teflon needle. The readings of interfacial tension were taken until a constant reading was obtained. The instrument was calibrated with pure water. All measurements were carried out at 25.00 ± 0.01°C.

The steady-state pyrene fluorescence measurements were carried out on a Perkin–Elmer Spectrofluorometer (Model LS 50 B, UK) with an excitation wavelength of 335 nm. The concentration of pyrene used in all measurements was approximately equal to 10^{-6} mol dm^{-3}. The ratio of the intensity of the pyrene emission (I_1/I_3) was used for evaluating the polarity of the environment in which the pyrene was solubilized. The excitation and emission slits were kept constant at 3 nm.

Results and discussion

Critical micelle concentration (cmc)

Spontaneous adsorption of surfactant from the bulk to the air-water interface leads to decrease in the interfacial tension (γ) with increasing in the bulk concentration (C) of surfactant. At a certain concentration, the γ versus logarithm of bulk concentration (C) plots shows a distinct break point corresponding to the cmc of the surfactant with the saturated interfacial monolayer of adsorbed surfactant. Figure 1 shows the γ-log C plots of SDS + DHPC system over the whole mole fraction range of SDS studied. The values of cmc obtained from the break point are summarized in Table 1, while the plots of fluorescence intensity ratio (I_1/I_3) and an value of cmc of SDS + DHPC system are shown in Fig. 2 and Table 2, respectively. The I_1/I_3 variation of pyrene demonstrates the bulk micellar properties of surfactants. A significant decrease in the I_1/I_3 values with an increase in the concentration of surfactants indicates that the

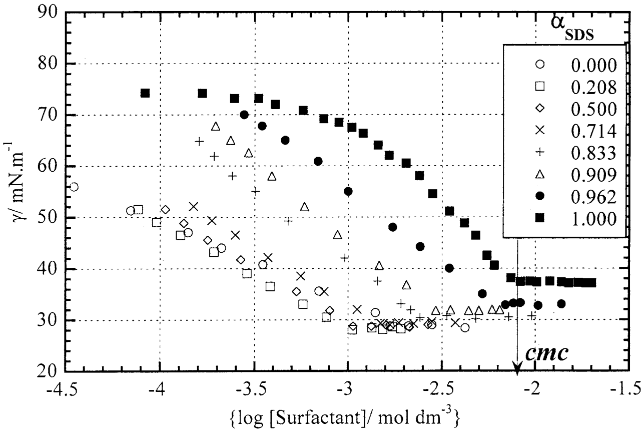

Fig. 1 Plot of interfacial tension (γ) versus log C of SDS + DHPC mixtures in pure water

Table 1 Micellar parameters for SDS + DHPC mixed systems obtained from interfacial study

α_{SDS}	cmc* (mM)	cmc (mM)	x_1	β
0.000	1.70	1.70	0.00	–
0.208	2.04	1.06	0.29	−4.72
0.500	2.81	1.06	0.39	−4.83
0.714	3.91	1.49	0.45	−4.11
0.833	5.02	2.40	0.51	−3.07
0.909	6.08	2.93	0.58	−3.29
0.962	7.00	6.90	–	–
1.000	8.20	8.20	1.00	–
				Average$\beta = -4.00$

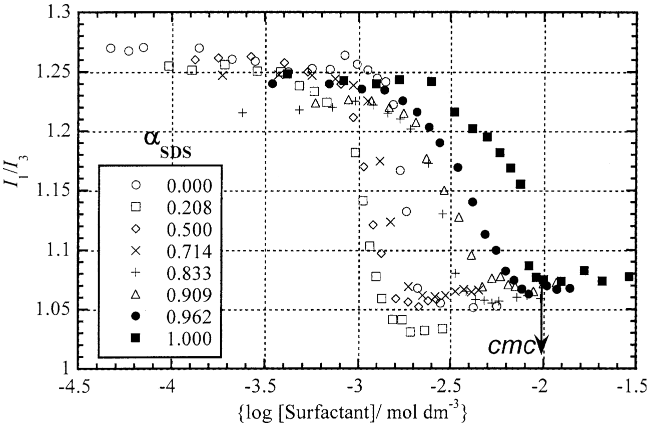

Fig. 2 Plot of fluorescence intensity (I_1/I_3) versus log C of SDS + DHPC mixtures in pure water

pyrene probe is taken up into the interior of the micelles. The intersection point between the end of the reversed sigmoidal curve and subsequent linear stretch gives the value of cmc. The cmc values obtained from the interfacial tension and the fluorescence intensity are slightly differ from each other, indicating that method dependent variations are apparent, as have also been seen for the pure and binary mixtures of cationic surfactants [20]. The cmc values have also been compared with the reported literature value [5, 21].

Table 2 Micellar parameters for SDS + DHPC mixed systems obtained from fluorescence study

α_{SDS}	cmc* (mM)	cmc (mM)	x_1	β
0.000	2.10	2.10	0.00	–
0.208	2.48	1.54	0.27	−3.63
0.500	3.30	1.59	0.38	−3.55
0.714	4.40	2.00	0.46	−3.33
0.833	5.50	4.40	0.55	−1.02
0.909	6.40	5.30	0.66	−1.00
0.962	7.30	7.00	–	–
1.000	8.20	8.20	1.00	–
				Average$\beta = -2.50$

Adsorption at the air-water interface

The adsorption behavior of mixtures of soluble surfactants at the interface and its consequent effect on interfacial properties varies significantly with the two surfactant's relative concentration. At surfactant concentration far below the cmc, the interface is only sparsely covered by surfactant molecules and a significant amount of bulk-like water remains at the interface. As the concentrations of surfactant and phospholipids increase, they orient at the air-water interface and substantially decrease the interfacial tension. The interfacial adsorption per unit area at various concentrations can be calculated with the help of the Gibbs adsorption equation. The surface pressure π (π = surface tension of pure water (γ_0)—surface tension of surfactant solution (γ)) is shown by the following equation

$$d\pi = \sum \Gamma_i RT \, d\ln a_i \tag{1}$$

where Γ_i and a_i are the surface excess and the activity of the ith component at temperature T, respectively. For a constant composition of the surfactant mixture with total concentration C,

$$\Gamma = \frac{1}{2.303RT}\left[d\pi(d\log C + d\log\gamma)^{-1}\right] \tag{2}$$

For the present dilute surfactant solution, the activity coefficient, $d\log\gamma_{\pm}$, is neglected [5, 20].

The maximum surface excess (Γ_{max}) and the minimum area per molecule of a surface-active compound (A_{min}) can be estimated from the relation:

$$\Gamma_{max} = \frac{1}{2.303RT} \cdot \lim_{C \to cmc}\left[d\pi(d\log C)^{-1}\right] \tag{3}$$

and

$$A_{min} = 10^{18}/N\Gamma_{max} \tag{4}$$

where N is Avogadro's number. The values of Γ_{max} and A_{min} have been calculated for the pure and binary combinations of SDS and DHPC from the least square fits of slopes of π versus log C plot. For our system consisting of a zwitterionic and an ionic component, the surface excess values were calculated by considering the factor "1/2.303" in the Eqs. (2) and (3) for the pure zwitterionic and binary mixtures at whole mole fraction range due to the decrease in the concentration of ionic surfactant at the interface, as compared to pure ionic SDS, where the coefficient "4.61" is used [5, 6, 20, 22]. The results are presented in Table 3. Figure 1 indicates that DHPC is more surface active than SDS, since its γ values are considerable lower than those of SDS. Further, it is evident from Table 3 that Γ_{max} for the mixtures gradually increases with the mole fraction of SDS. This might reflect on the formation of compact

Table 3 Interfacial thermodynamic parameters for SDS + DHPC mixed system

α_{SDS}	Γ_{max} x 10^6 (mol m^{-2})	A_{min}(nm^2)	π_{cmc} (mNm^{-1})	-$\Delta G^o{}_m$ (kJ mol^{-1})	β^σ	x_1^σ
0.000	2.76	0.60	43.2	25.7	–	0.00
0.208	3.61	0.46	44.0	26.9	−4.80	0.18
0.500	3.88	0.43	43.3	26.9	−5.22	0.28
0.714	3.95	0.42	42.7	26.0	−4.72	0.33
0.833	4.95	0.34	41.6	24.9	−2.49	0.32
0.909	5.35	0.31	40.2	24.0	−2.29	0.39
0.962	4.84	0.34	39.1	22.3	−0.29	0.50
1.000	3.13	0.54	34.6	21.9	–	1.00
						Average $\beta = -3.30$

interfacially adsorbed mixed surfactant monolayer because of attractive interactions or van der Waals forces between the double hydrophobic tail of phospholipid and the single tail of surfactant. On the other hand, the A_{min} values are in the reverse order.

Thermodynamics of micellization

The standard free energy of micellization ($\Delta G^o{}_m$) for SDS, DHPC, and their binary mixtures was calculated by applying the equation [5, 23]:

$$\Delta G^o_m = RT \ln X_{cmc} \tag{5}$$

Unless the cmc is very high, $\mathbf{X}_{cmc}$ can be approximated as an cmc/ω, where ω is the molar concentration of solvent. Using cmc values, $\Delta G^o{}_m$ values for the pure and mixed systems are computed and listed in Table 3. It has been observed that all the $\Delta G^o{}_m$ values are negative, that is, energetically favorable. The $\Delta G^o{}_m$ value of DHPC is more negative than that of SDS, probably because DHPC with two C-7 chains is more hydrophobic than SDS with a single C-12 chain. Further, the $\Delta G^o{}_m$ values decrease between 0 and 0.5 mole fraction of SDS, and subsequently increase slightly. Micelle formation may be more favorable at low to intermediate SDS concentrations due to the compactness of the micelle. Above 0.5 mole fraction of SDS, electrostatic repulsion increased which disfavors micelle formation. The standard free energy of interfacial adsorption ($\Delta G^o{}_{ad}$) is more negative for DHPC than SDS indicating that the DHPC has a tendency to exhibit greater attractive hydrophobic interaction due to the presence of double hydrocarbon tails in their structure than SDS, (data not shown) [24, 25].

Synergistic behavior of mixed micelle and mixed monolayer

The mixed cmc values for SDS and DHPC are shown in Fig. 3 and Tables 1 and 2. In order to explore whether the mixed micelles show ideal behavior or not, quantitative information about the micellar aggregates formation can be obtained by using the pseudo-phase separation model [16, 17]. The model relates the ideal mixed cmc* with the experimental cmc of the pure components by using the following equations:

$$C_1 = x_1 \text{cmc}_1 = \alpha_1 \text{cmc}^* \tag{6}$$

and

$$C_2 = (1 - x_1)\text{cmc}_2 = (1 - \alpha_1)\text{cmc}^* \tag{7}$$

Equation (8) can be obtained after combining Eqs. (6) and (7)

$$\frac{1}{\text{cmc}^*} = \frac{\alpha_1}{\text{cmc}_1} + \frac{(1 - \alpha_1)}{\text{cmc}_2} \tag{8}$$

The α_1 and x_1 are the mole fractions of surfactant 1 (SDS) in the total mixed solute and in the mixed micelle, respectively. The cmc_1, cmc_2, C_1, and C_2 are the critical micellar concentrations and the overall concentrations in the micelle formation of components 1 and 2, respectively.

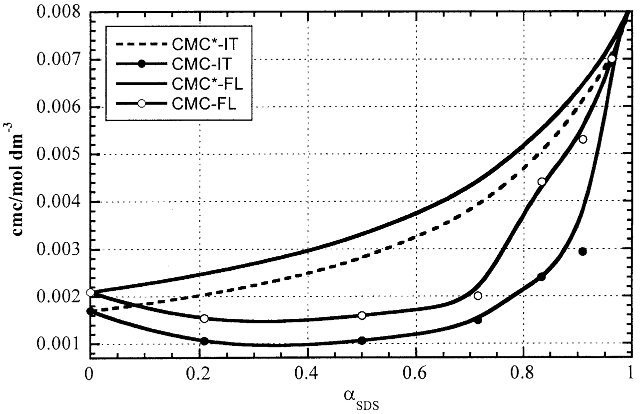

Fig. 3 Plot of mixed critical micelle concentration (cmc) versus α_{SDS} for SDS + DHPC mixtures in pure water. Predicted cmc* (line), Experimental cmc (*Black circles*, interfacial data), (*white circles*, fluorescence data)

Since the present binary mixture is a combination of ionic surfactant + zwitterionic lipid, a nonideal behavior is expected due to significant differences between their head groups and hydrophobic tail. The cmc* values thus calculated using Eq. (8) have also been listed in Tables 1 and 2. The mixed cmc values of Tables 1 and 2 are plotted in Fig. 3 along with the ideal cmc*. The experimental cmc value shows negative departure from the ideal cmc* over the whole mixing range. This result demonstrates that a significant degree of synergism exists in the mixed micelle formation between SDS and DHPC due to favorable interactions between the dissimilar monomers.

Since the mixing behavior of present binary mixtures shows synergism, these results have been further confirmed by using the regular solution approximation [18]. The x_1 can be iteratively computed from the following equation:

$$\frac{x_1^2 \ln(\mathrm{cmc}\alpha_1/\mathrm{cmc}_1 x_1)}{(1-x_1)^2 \ln(\mathrm{cmc}(1-\alpha_1)/\mathrm{cmc}_2(1-x_1))} = 1 \tag{9}$$

The x_1 values are listed in Tables 1 and 2. From x_1 values, the interaction parameter, β, can be computed by the following equation:

$$\beta = \frac{\ln\left(\frac{\mathrm{cmc}\alpha_1}{\mathrm{cmc}_1 x_1}\right)}{(1-x_1)^2} \tag{10}$$

The β value demonstrates the extent of interaction between the two surfactants which lead to the deviation from ideal behavior. The negative and positive β values suggest attractive and repulsive interactions between two surfactants in a mixed micelle, respectively. For the ideal mixing, the β value should be close to zero. The β values computed for the SDS + DHPC are given in Tables 1 and 2 and shows that these values are negative at whole mixing range. The average β values calculated from interfacial and fluorescence studies are −4.00 and −2.50, respectively. These values are negative which suggest that mixed micelle formation takes place owing to favorable (attractive) interaction between unlike components. Interestingly, β values becomes less negative with the increase in the mole fraction of SDS indicating weaker synergism. The induction of SDS increases the polarity of the mixed micelle which leads to the formation of loose aggregates and weakens the attractive interactions [26].

The mixed micellar regular solution theory can be extended to the mixed monolayer. In order to evaluate the interaction parameter,β^σ, in the mixed monolayer, the analogous Eqs. (11) and (12) can be derived at a constant interfacial tension [6, 27]

$$\frac{x_1^2 \ln(C_{12}\alpha_1/C_1^0 x_1)}{(1-x_1)^2 \ln(C_{12}(1-\alpha_1)/C_2^0(1-x_1))} = 1 \tag{11}$$

and

$$\beta^\sigma = \frac{\ln\left(\frac{C_{12}\alpha_1}{C_1^0 x_1}\right)}{(1-x_1)^2} \tag{12}$$

where x_1 is the mole fraction of surfactant 1 in the total surfactant in the mixed monolayer at the interface. C_1^0, C_2^0, and C_{12} are the solution phase molar concentrations of individual surfactants 1 and 2 and their mixtures, required to produce given interfacial tension. The β^σ values are evaluated from the above equation at 45 mN.m^{-1} of the interfacial tension and listed in Table 3. These values are also negative indicating that synergism occurs even in the mixed monolayer formation. Interestingly, β^σ values also become weaker with an increase in the amount of SDS due to the electrostatic repulsive forces of ionic polar head group of the surfactant at the interface, which destabilize the mixed monolayer. The average β^σ value evaluated is −3.30. This value also shows nonideal behavior [6, 8, 27]. Thus if we compare the average of β^σ with β, it is very hard to discriminate whether interactions are more pronounced in the mixed monolayer or mixed micelle, since both values do not show much difference. A comparison of β^σ with β obtained from fluorescence shows that interactions are slightly favorable at the interface, since the fluorescence method is considered to be more sensitive to ongoing changes in the bulk solution. The x_1^σ values at 45 mNm^{-1} have also been obtained and shown in Table 3.

On the other hand, in the mixed micelle formation, the existence of synergism in mixtures containing two surfactants depends not only on the strength of the interaction between them, but also on relevant properties of the individual surfactant component of the binary amphiphilic mixture. Thus, there are condition required for the synergism [6, 8, 27]; (1) β should be negative (2)$|\beta| > |\ln \mathrm{cmc}_1/\mathrm{cmc}_2|$. Both conditions are satisfied in the present binary mixtures for interfacial data. The degree of synergism in mixed micelle formation is measured by the maximum decrease in molar concentration of the mixed surfactant in comparison to that of individual components, i.e.(cmc_1-$\mathrm{cmc}_{12,\min}$)/ cmc_1 or 1 − ($\mathrm{cmc}_{12,\min}/\mathrm{cmc}_1$), where $\mathrm{cmc}_{12,\min}$ is the minimum cmc of the mixed surfactants. The ratio $\mathrm{cmc}_{12,\min}/\mathrm{cmc}_1$ is given by the relationship:

$$\frac{\mathrm{cmc}_{12,\min}}{\mathrm{cmc}_1} = \exp\left(\frac{[\beta - \ln(\mathrm{cmc}_1/\mathrm{cmc}_2)]^2}{4\beta}\right) \tag{13}$$

The greater the value of 1—($\mathrm{cmc}_{12,\min}/\mathrm{cmc}_1$), the greater the degree of synergism. These values have been calculated (data not shown) and are more favorable from 0.87 up to the equimolar mixture, which suggest that maximum degree of synergism exist at equimolar proportion. This might be the reason that both γ and the intensity the ratio lying lower, that is up to equimolar proportion indicate the presence of aggregates with high hydrophobicity. In order to confirm this result, we have also evaluated the point of maximum synergism in mixed micelle formation. This method states that the minimum mixed concentration required to produce synergism and the mole fraction of the surfactant 1 in the solution phase equals its mole fraction in the mixed micelle [6, 8, 28]. This behavior is shown in Fig. 4 marked by an arrow where the micellar mole fraction (X_1) lies close to the bulk mole fraction (α_1). Also, the cmc at the point of maximum synergism, (the minimum total mixed surfactant concentration in the solution phase required for mixed micelle formation) is given by the following relationship:

$$\mathrm{cmc}_{12,\mathrm{min}} = \mathrm{cmc}_1 \exp\left\{\beta\left[\frac{\beta - \ln(\mathrm{cmc}_1/\mathrm{cmc}_2)}{2\beta}\right]^2\right\} \quad (14)$$

These values have been computed and found to be lower at $\alpha_{\mathrm{SDS}} = 0.5$ which is 9.9×10^{-4} mol dm^{-3}.

From the interaction parameter β, and mole fraction of surfactant 1 in the mixed solute (α_1), the micellar mole fraction of component (1) in the mixed micelle has been computed and shown in Fig.4. The solid line corresponds to ideal behavior ($\alpha_1 = X_1$). The X_1 values were found to deviate from positive to negative from ideal behavior, suggesting a decrease in the fraction of SDS in the mixed micelle in SDS rich regions. This behavior is attributed to the electrostatic repulsive forces between the polar ionic head groups of SDS which inhibits its monomers from entering the mixed micelle. Similar behavior has been observed for the binary mixture of cationic and nonionic surfactants where electrostatic repulsion becomes stronger at the high mole fraction of the cationic component [28].

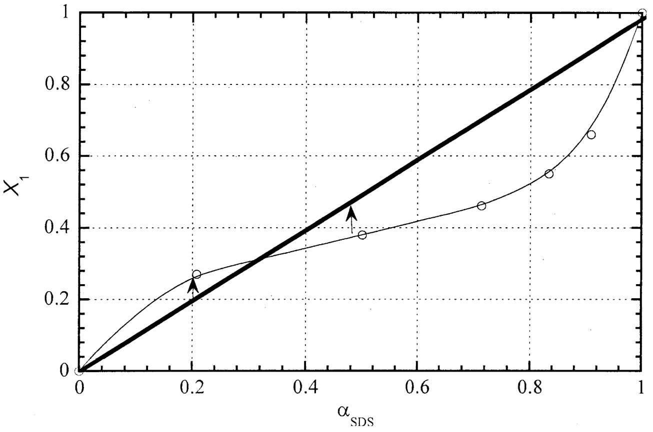

Fig. 4 Plot of mole fraction of SDS in the mixed micelle (X_1) versus mole fraction in the bulk (α) for SDS + DHPC mixtures in pure water (fluorescence data). *Solid line* predicts the ideal value, *white circles* the experimental value

Conclusion

The present work deals with interfacial and micellar study of SDS and DHPC in an aqueous medium. From the interfacial study various parameters such as Γ_{max} or A_{min} have been evaluated. The negative values of $\Delta G^{\mathrm{o}}{}_{\mathrm{m}}$ shows that micelle formation is energetically favorable. A significant negative departure from ideal cmc* has been observed, indicating the existence of synergism between two unlike components which led to non-ideal mixing of the binary mixture. The synergism becomes weaker with the increase in the amount of SDS due to electrostatic repulsion between the ionic species of SDS or an increase in the polarity of the mixed micelle which produces loose aggregates with less surface charge density. The micellar mole fraction obtained from regular solution approximation method indicates that mixed micelles are less rich in SDS, even in the SDS rich region, and these values are lower than ideal behavior. The negative values of β and β^{σ} also suggest that mixed micelle and monolayer formation take place due to the favorable interaction of amphiphilic molecules, respectively. The incorporation of SDS into the mixed micelle weakens synergism between the amphiphilc molecules.

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