

Temperature Dependence of Micellization Properties of Binary Mixtures of Sodium Decylsulphate with Sodium Dodecylsulphate in Water

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Conductance measurement method has been used to determine the critical micelle concentration (CMC) and counterion binding constant values of binary mixtures of sodium decylsulphate (SDeS) with sodium dodecylsulphate (SDS) in water at four different temperatures ranging from 298 K to 313 K at 5 K intervals. Rubingh's model has been used to calculate the mixed micellization properties *viz.* mixed micelle composition, activity coefficient of the components, and mutual interaction parameter between the components. Variation of these mixed micellization properties with temperature has been studied. The studied binary surfactant mixtures show attractive interaction between the components and it decreases with increase in temperature, which is an interesting trend. Thermodynamic parameters of mixed micellization have also been calculated. The enthalpy (ΔH_{mic}°) and entropy (ΔS_{mic}°) of micellization compensate each other.

Keywords: Critical micelle concentration, Counterion binding constant, Sodium decylsulphate, Sodium dodecylsulphate, Mixed micelle.

INTRODUCTION

Surface-active agents or surfactants in solution show two interesting phenomena *viz.* (i) adsorption at the interface and (ii) micellization at critical micelle concentration (CMC). The presence of both hydrophobic and hydrophilic part gives a surfactant molecule its amphiphilic behaviour. Owing to their amphiphilic property surfactants are found used in various fields including detergency, drug-delivery, emulsion stabilization, *etc.* It is well established that mixtures of surfactants perform better than single surfactant systems. Hence, many formulations developed for application in various fields consisting of surfactant mixtures are found. Mixed surfactant systems have both academic and industrial importance and have drawn attentions of many researchers [1-13]. It may be mentioned that even in recent times, mixed surfactant systems are widely studied [14-16].

Effect of temperature on the micellization properties of single surfactant systems have been studied by many researchers [17-29]. Therefore, thermodynamic parameters of micellization of single surfactant systems are available in literature. However, systematic studies on the effect of temperature on micellization properties of mixed surfactant systems are scanty and hence

the thermodynamic parameters too. Further, amongst the various types of surfactants, sodium alkylsulphate surfactants are the most extensively studied systems. In a previous study [30], it has been reported that the mutual attractive interaction in the binary mixtures of sodium tetradecylsulphate (STS) with sodium decylsulphate (SDeS) or sodium dodecylsulphate (SDS) increases with increase in temperature. In view of the above considerations and to further substantiate the above observation, effect of temperature on the micellization properties of binary mixtures of SDeS with SDS in water medium has been studied in the present study though the same mixed surfactant systems have already been studied by conductivity measurement method at 298 K [31].

In this present study, conductance measurements were conducted at four different temperatures ranging from 298 K to 313 K at 5 K intervals to determine the CMC and counterion binding constant values at each temperature both for pure and binary mixed systems. Rubingh's model [32,33] has been used to calculate mixed micelle composition, activity coefficient of the components and mutual interaction between the components in the mixed micelle. Variation of CMC with temperature has been considered to calculate the thermodynamic parameters for both pure and mixed systems. Moreover, thermodynamic

parameters of mixed micellization of the binary mixed surfactant systems studied are also presented.

EXPERIMENTAL

The anionic surfactants, sodium decylsulphate (SDeS) and sodium dodecylsulphate (SDS) were purchased from Sigma-Aldrich Co. Ltd. and used without further purification. Double distilled water with specific conductance (κ) less than $2 \mu\text{S cm}^{-1}$ at 298 K was used for the preparation of all the solutions. A binary mixed surfactants solution of a particular composition was prepared by weighing required amounts of the two surfactants and then dissolving them together. Concentrated surfactant solution in small amounts was progressively added to a known quantity of double distilled water using an Eppendorf pipette. Conductance measurements were made after each addition of the concentrated surfactant solution and after equilibrating using digital conductivity TDS meter (Model 308, Systronics) with a dip type conductivity cell of cell constant equal to 1 cm^{-1} under controlled temperature conditions using a water bath thermostat. A Shimadzu balance (Model ATX224) was used for measurements of weights whenever required.

RESULTS AND DISCUSSION

Critical micelle concentration (CMC): Representative plots of experimental values of specific conductance (κ) versus total surfactant concentration (C) at different temperatures for SDeS + SDS mixed systems, (a) $\alpha_{\text{SDS}} = 0.25$ and (b) $\alpha_{\text{SDS}} = 0.75$ are shown in Fig. 1. Similar plots were obtained for both pure and equimolar mixture. From these plots, CMC values have been determined from the point of intersection of the two straight lines in the pre-micellar and post-micellar regions. The CMC values thus determined are listed in Table-1. At a particular temperature, for the pure aqueous surfactant solutions, CMC value of SDeS is greater than that of SDS. The

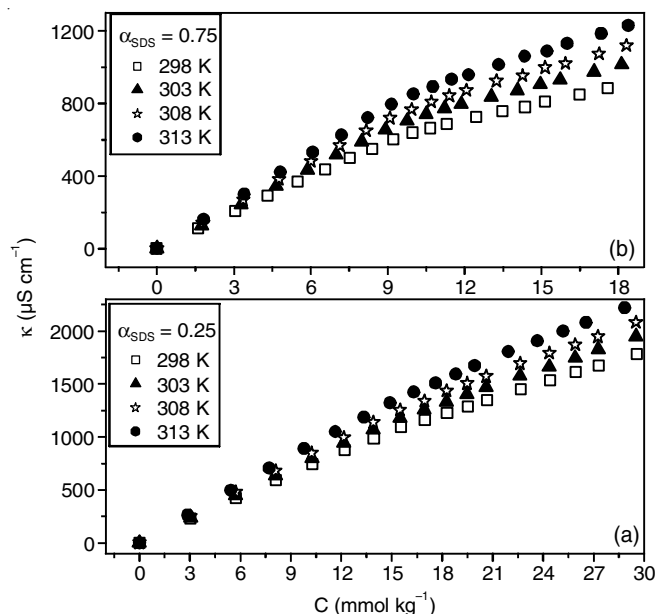


Fig. 1. Representative plots of specific conductance (κ) against total surfactant concentration (C) at different temperatures for SDeS + SDS mixed binary systems (a) $\alpha_{\text{SDS}} = 0.25$ (b) $\alpha_{\text{SDS}} = 0.75$

observed CMC values for the pure aqueous surfactant solutions in the temperature range studied are comparable with previously reported [25] CMC values determined using conductivity measurement. The CMC values for both pure and binary mixed systems increase with increase in temperature in the investigated temperature range except for SDeS with a minimum at 303 K. Such a minimum has been reported earlier for sodium alkylsulphate systems [24]. Generally, for both ionic and non-ionic surfactants, there exist a minimum in the CMC-temperature curve and the temperature of minimum CMC increases with decrease in hydrophobicity of surfactants [26]. This may be the reason for SDS not showing a minimum CMC in the temperature range studied.

TABLE-1
VALUES OF CMC, β_c , $x_{\text{SDS}}^{\text{Rb}}$, β_m , f_{SDS} and f_{SDeS} FOR SDeS + SDS SYSTEMS IN WATER AT DIFFERENT TEMPERATURES

α_{SDS}	Temp. (K)	CMC (mmol kg ⁻¹)	β_c	$x_{\text{SDS}}^{\text{Rb}}$ (x_{SDS}^*)	β_m	f_{SDS}	f_{SDeS}
0	298	33.0	0.54	—	—	—	—
	303	32.8	0.53	—	—	—	—
	308	33.0	0.53	—	—	—	—
	313	33.4	0.52	—	—	—	—
0.25	298	14.85 (18.53 ^a)	0.30	0.555 (0.579)	-0.900	0.836	0.758
	303	15.08 (18.61)	0.29	0.552 (0.574)	-0.855	0.842	0.770
	308	15.57 (18.92)	0.28	0.550 (0.570)	-0.791	0.852	0.787
	313	16.18 (19.41)	0.28	0.547 (0.564)	-0.736	0.860	0.802
0.50	298	10.70 (12.88)	0.36	0.723 (0.805)	-1.022	0.925	0.586
	303	10.79 (12.99)	0.34	0.721 (0.802)	-1.017	0.924	0.589
	308	11.01 (13.26)	0.33	0.719 (0.799)	-1.012	0.923	0.593
	313	11.59 (13.68)	0.32	0.722 (0.795)	-0.901	0.933	0.625
0.75	298	9.50 (9.87)	0.51	0.895 (0.925)	-0.469	0.995	0.687
	303	9.65 (9.98)	0.51	0.897 (0.924)	-0.413	0.996	0.717
	308	9.90 (10.21)	0.50	0.898 (0.923)	-0.380	0.996	0.736
	313	10.25 (10.56)	0.49	0.897 (0.921)	-0.362	0.996	0.747
1.00	298	8.0	0.60	—	—	—	—
	303	8.1	0.59	—	—	—	—
	308	8.3	0.58	—	—	—	—
	313	8.6	0.57	—	—	—	—

^aCMC value obtained from Clint's equation.

The CMC of a binary mixed surfactants (C_{mix}) is related to the bulk composition (α) and CMC's of components (C_{SDS}) and (C_{SDeS}) by the relation [32,33]:

$$\frac{1}{C_{\text{mix}}} = \frac{\alpha_{\text{SDS}}}{f_{\text{SDS}} C_{\text{SDS}}} + \frac{\alpha_{\text{SDeS}}}{f_{\text{SDeS}} C_{\text{SDeS}}} \quad (1)$$

$$f_i = \frac{\alpha_i C_{\text{mix}}}{x_i C_i} \quad (2)$$

In eqn. 2 x_i and f_i refer to mole fraction and activity coefficient of the i^{th} component ($i = \text{SDS}$ or SDeS) in the mixed micelle, respectively. For an ideal mixed micelle ($f_i = 1$), eqn. 1 reduces to the Clint's equation [34]:

$$\frac{1}{C_{\text{mix}}} = \sum \frac{\alpha_i}{C_i} \quad (3)$$

From Table-1, it is observed that the experimental CMC values of the binary mixtures are less than the ideal CMC values, which were obtained using eqn. 3, thereby indicating synergism in the studied temperature range.

Mixed micelle composition: Rubingh described the non-ideality in the mixed micelle by employing the regular solution approximation [32,33]. According to this approximation, non-ideality in the mixed micelle is described in terms of the mutual interaction parameter (β_m) and is related to the activity coefficient by the following relation:

$$\beta_m = \frac{\ln f_{\text{SDS}}}{(1-x_{\text{SDS}})^2} = \frac{\ln f_{\text{SDeS}}}{x_{\text{SDS}}^2} \quad (4)$$

On substituting for the activity coefficients in eqn. 4, we get the expression:

$$x_{\text{SDS}}^2 \ln \frac{\alpha_{\text{SDS}} C_{\text{mix}}}{x_{\text{SDS}} C_{\text{SDS}}} = (1-x_{\text{SDS}})^2 \ln \frac{(1-\alpha_{\text{SDS}}) C_{\text{mix}}}{(1-x_{\text{SDS}}) C_{\text{SDeS}}} \quad (5)$$

At a particular bulk composition α_{SDS} , values of mole fraction of SDS in the mixed micelle x_{SDS} at different temperatures were computed from eqn. 5 by using an iterative method. The x_{SDS} values obtained in this manner are also given in Table-1 as $x_{\text{SDS}}^{\text{Rb}}$. Under the condition of ideality ($f_i = 1$), x_{SDS} takes the form

$$x_{\text{SDS}} = \frac{\alpha_{\text{SDS}} C_{\text{SDeS}}}{\alpha_{\text{SDS}} C_{\text{SDeS}} + \alpha_{\text{SDeS}} C_{\text{SDS}}} \quad (6)$$

The ideal values of x_{SDS} calculated employing eqn. 6 are given in Table-1 as x_{SDS}^* . It is observed that in the mixed systems studied, the mixed micelle is enriched with the surfactant component with lower CMC value (higher homologue). Similar observation of enrichment of the mixed micelle with the surfactant component with lower CMC was made in SDeS + SDS system studied earlier [31]. In general, the enrichment of the mixed micelle by SDS in the present study remains practically constant with increase in temperature in the studied temperature range.

The activity coefficient f_i values at different temperatures were calculated using eqn. 2 and by employing the x_{SDS} values which were computed using eqn. 5. The calculated values of f_{SDS} and f_{SDeS} are given in Table-1. Activity coefficient represents the effect and contribution of an individual component

in the mixed micelle. In the mixed systems studied, the f_{SDS} values are high and more than that of f_{SDeS} , indicating that the SDS component in the mixed micelle is close to its standard state. In general, the activity coefficient of SDS remains practically constant while that of SDeS show appreciable increase with increase in temperature in the studied temperature range.

The values of the mutual interaction parameter β_m were determined using eqn. 4 and at different temperatures are given in Table-1. It is observed that β_m values are negative indicating attractive interaction between the anionic surfactant components. A more negative value of β_m indicates stronger attractive interaction between the components. It may be mentioned that strongest attractive interaction between the components occurs at equimolar composition of the mixed system. In general, the attractive interaction between the components in the SDeS + SDS mixed systems is found to decrease with increase in temperature, which is an opposite trend to that observed for STS + SDeS and STS + SDS mixed systems [30]. In all the three mixed systems, SDeS + SDS, STS + SDeS and STS + SDS, the hydrophilic head group is the same and hence, the observed interesting opposite trend can be attributed to the hydrophobic tail group interaction. At this moment it seems increase in temperature brings about some compactness in the micelles, which can lead to either (i) increase in attractive interaction due to stronger hydrophobic tail-tail interaction or (ii) decrease in attractive interaction due to weaker hydrophobic tail-tail interaction.

Counterion binding constant: The slope ratio method, being simple and reliable, has been used to evaluate the counterion binding constant (β_c) values of both pure and mixed surfactant systems. In this method, β_c is calculated using the relation:

$$\beta_c = 1 - \frac{S_2}{S_1} \quad (7)$$

where, S_1 and S_2 are the slopes of the specific conductance (κ) versus surfactant concentration (C) plots in the pre and post micellar regions, respectively. The calculated β_c values are given in Table-1. At a particular temperature, for pure aqueous surfactant solutions, β_c value of SDS is greater than that of SDeS. It may be mentioned that the β_c values for pure SDeS and SDS in the temperature range studied are close to the values reported earlier using conductivity method at similar temperatures [17]. The β_c value decreases slightly with increase in temperature which may be due to decrease in the aggregation number of micelle [35]. However, β_c has a weak dependence on temperature. It may also be pointed out that the β_c values of the mixed micelles are lower than those of pure micelles. The low β_c values for mixed micelles shows that the effective surface charge density is less in the mixed micelles than that in the pure micelles. Similar observation of low β_c values for mixed micelles has been reported for equimolar; CTAB + TTAB, CTAB + DTAB and TTAB + DTAB binary cationic mixed systems [2].

Thermodynamic parameters of micellization: The standard Gibbs free energy of micellization per mole of surfactant ($\Delta G_{\text{mic}}^{\circ}$) of both pure and the mixed surfactant systems has been obtained using the following relation [26,36]:

$$\Delta G_{\text{mic}}^{\circ} = (1 + \beta_c) RT \ln X_{\text{cmc}} \quad (8)$$

where, R is the gas constant ($8.314 \text{ J K}^{-1} \text{ mol}^{-1}$), T is absolute temperature and X_{cmc} is the CMC in mole fraction unit. The corresponding enthalpy change of micellization, $\Delta H_{\text{mic}}^{\circ}$, is

$$\Delta H_{\text{mic}}^{\circ} = -RT^2 \left[(1 + \beta_c) \left(\frac{\partial \ln X_{\text{cmc}}}{\partial T} \right)_p + \ln X_{\text{cmc}} \left(\frac{\partial \beta_c}{\partial T} \right)_p \right] \quad (9)$$

Since the dependence of β_c on temperature is small in the temperature range investigated, eqn. 9 becomes

$$\Delta H_{\text{mic}}^{\circ} = -RT^2 \left[(1 + \beta_c) \left(\frac{\partial \ln X_{\text{cmc}}}{\partial T} \right)_p \right] \quad (10)$$

$\Delta H_{\text{mic}}^{\circ}$ values were calculated using eqn. 10. For this purpose, $\ln X_{\text{cmc}}$ was plotted against T and the slopes determined at each

temperature is taken as $\left(\frac{\partial \ln X_{\text{cmc}}}{\partial T} \right)_p$. Once $\Delta G_{\text{mic}}^{\circ}$ and $\Delta H_{\text{mic}}^{\circ}$

have been calculated, the entropic contribution has been obtained using the following relation

$$\Delta G_{\text{mic}}^{\circ} = \Delta H_{\text{mic}}^{\circ} - T\Delta S_{\text{mic}}^{\circ} \quad (11)$$

The thermodynamic parameters of micellization, $\Delta G_{\text{mic}}^{\circ}$, $\Delta H_{\text{mic}}^{\circ}$ and $\Delta S_{\text{mic}}^{\circ}$ for the investigated pure and mixed surfactant systems are given in Table-2. It is observed that for both pure and mixed surfactant systems, $\Delta G_{\text{mic}}^{\circ}$ becomes more negative with increase in temperature. It is also evident that the enthalpy of micellization becomes more negative and the associated London-dispersion interaction plays a more predominant role as the temperature increases. The entropic contribution of micelle formation, $T\Delta S_{\text{mic}}^{\circ}$, has positive values and it decreases with rise in temperature. This shows that the micellization process, either in pure or in mixed state, is entropy controlled at lower temperatures while at higher temperatures, it is enthalpy controlled.

The phenomenon of enthalpy-entropy compensation which has been observed in a variety of processes, including the micelle

formation of surfactants [26,27,37,38], is reflected by a linear correlation between the enthalpy change and the entropy change and is expressed as follows:

$$\Delta H_{\text{mic}}^{\circ} = \Delta H_{\text{mic}}^* - T_c \Delta S_{\text{mic}}^{\circ} \quad (12)$$

In order to analyze the enthalpy-entropy compensation phenomenon, the plots of $\Delta H_{\text{mic}}^{\circ}$ versus $\Delta S_{\text{mic}}^{\circ}$ i.e. the so-called compensation plot have been considered. According to the conceptual scheme of compensation phenomenon proposed by Lumry and Rajender [38], micelle formation process can be divided into (i) desolvation part and (ii) chemical part. The slope of the compensation plot, T_c , known as the compensation temperature, provides a measure of the desolvation part of micellization. The intercept, ΔH_{mic}^* , gives information of the solute-solute interaction and reflects the effectiveness of the chemical part of the micelle formation. The compensation plots for SDeS + SDS mixed systems are shown in Fig. 2. A linear correlation between the enthalpy and entropy of micellization has been observed for all the surfactant systems studied. The values of ΔH_{mic}^* and T_c both for pure and mixed surfactants obtained from the compensation plots are given in Table-2. The ΔH_{mic}^* value becomes less negative on initial addition of SDS to SDeS but on further addition it becomes more negative. It has been reported that ΔH_{mic}^* decreases (becomes more negative) with an increase in the alkyl chain length of surfactants in a homologous series and a more negative value of ΔH_{mic}^* means greater stability of the structure of the micelle [26]. Therefore, it may be inferred that the addition of higher homologue (SDS) to SDeS results in a more stable micelle by enhancing the effect of chemical part of micellization.

It has been reported that the desolvation part of micellization process is independent of alkyl chain length of surfactants in a homologous series [26]. In the present SDeS + SDS mixed systems, the T_c values are found to be $288 \pm 2 \text{ K}$ and is within the suggested range of 270-294 K for the water system [27].

TABLE-2
THERMODYNAMIC PARAMETERS OF MICELLIZATION FOR SDeS + SDS SYSTEMS IN WATER AT DIFFERENT TEMPERATURES

α_{SDS}	Temp. (K)	$\Delta G_{\text{mic}}^{\circ}$ (kJ mol ⁻¹)	$\Delta H_{\text{mic}}^{\circ}$ (kJ mol ⁻¹)	$T\Delta S_{\text{mic}}^{\circ}$ (kJ mol ⁻¹)	ΔH_{mic}^* (kJ mol ⁻¹)	T_c (K)
0	298	-28.35	2.132	30.48	-27.5	290
	303	-28.66	0.074	28.73		
	308	-29.11	-2.110	27.00		
	313	-29.34	-4.407	24.93		
0.25	298	-26.50	-2.236	24.26	-25.67	289
	303	-26.69	-4.564	22.12		
	308	-26.81	-7.006	19.80		
	313	-27.12	-9.639	17.48		
0.50	298	-28.83	1.250	30.08	-27.69	288
	303	-28.85	-3.120	25.73		
	308	-29.04	-7.706	21.33		
	313	-29.11	-12.516	16.60		
0.75	298	-32.45	-2.460	29.99	-31.38	287
	303	-32.93	-4.741	28.19		
	308	-33.16	-7.123	26.04		
	313	-33.34	-9.622	23.72		
1.00	298	-35.06	-1.607	33.46	-33.51	284
	303	-35.38	-4.452	30.93		
	308	-35.64	-7.448	28.19		
	313	-35.84	-10.594	25.25		

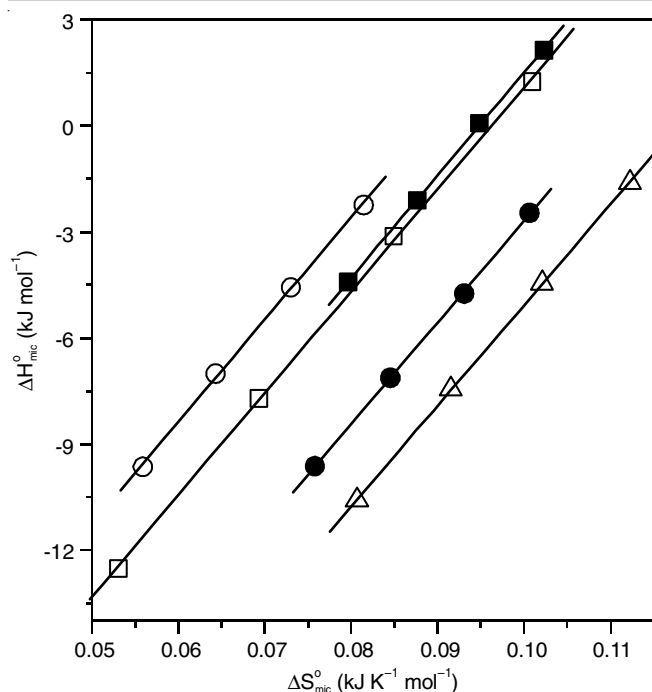


Fig. 2. Enthalpy-entropy compensation plots for SDeS + SDS mixed binary systems. (■) $\alpha_{\text{SDS}} = 0$; (○) $\alpha_{\text{SDS}} = 0.25$; (□) $\alpha_{\text{SDS}} = 0.50$; (●) $\alpha_{\text{SDS}} = 0.75$; (△) $\alpha_{\text{SDS}} = 1$

Conclusion

Micellization properties of binary mixtures of sodium decylsulphate (SDeS) with sodium dodecylsulphate (SDS) in water medium have been studied at temperatures ranging from 298K to 313K at 5 K intervals. In the investigated temperature range, CMC increases whereas counterion binding constant decreases slightly with increase in temperature. In the binary mixed systems of SDeS with SDS, the mixed micelles are enriched with the surfactant component having lower CMC (SDS) and the attractive interaction between the components decreases with increase in temperature. Micellization, both in pure and in mixed state, is found to be entropy driven at low temperatures whereas at high temperatures it is enthalpy driven. The enthalpy and entropy terms of micellization compensate each other, resulting in moderate decrease in the Gibbs energy.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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