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# Micellization of anionic surfactant-sodium dodecyl sulfate in presence of additive urea and acetamide in aqueous medium using clouding phenomenon

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## ABSTRACT

*The clouding phenomenon and micellization of anionic surfactant, sodium dodecyl sulfate have been studied by measuring the cloud points (CP) of the pure surfactant and mixed system with additives such as urea and acetamide. The combined effect of these additives on cloud point measurement was investigated. The cloud point of pure surfactant Sodium dodecyl sulphate (SDS) found to be increased with increased [SDS]. The cloud point of mixed system also shows same trends with increased [Urea] and [Acetamide]. This is mainly due to increased micelle concentrations. The influence of additive, urea on the cloud point of SDS is a clear indication that the phenomenon of clouding is associated with the different micelles coalescing. The phase separation results from micelle-micelle interaction, considering cloud point as threshold temperature of the solubility, the thermodynamic parameters of clouding process ($\Delta G^o_{C1}$, $\Delta H^o_{C1}$ and $\Delta S^o_{C1}$) have been evaluated using "Phase Separation Model" Kjellander et al model according to which clouding is an entropy dominated phenomena, have also been used for calculating the thermodynamic parameters. It is found that the overall clouding process is exothermic and $\Delta H^o_{C1} > T\Delta S^o_{C1}$ indicating that the process of clouding is guided by both enthalpy and entropy. This work supports the conjecture that the cloud point is critical phenomenon rather the growth of micelles.*

**Keywords:** Acetamide, Anionic surfactant, Cloud point, Phase separation model, Sodium dodecyl sulphate

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## INTRODUCTION

Surfactants are common place ingredients of many medical, home and personal care products. In solution, they self assemble into a range of well-defined structures that define their fundamental rheological dispersion and colloidal properties [1, 2]. The cloud point (CP) is an important property of surfactants and can be used in diverse applications [3-5] for example foam control in many industrial processes is important because foaming can limit the rate of these process and cause product instability. It has been found that the foam stability drops at a temperature near the CP [6]. A cloudy dispersion appears upon heating of non-ionic surfactant solutions [7-9]. The threshold temperature for clouding is known as the cloud point (CP). Linear alkyl benzene sulfonates (ABS), are an important class of anionic surfactants. They are one of the most commonly used commercial surfactants and usually exits as a mixture of alkyl chain homologues with a range of head group positional isomers.

The occurrence of CP phenomena is rarer for ionic surfactant; however, we have systematically studied the CP phenomenon in anionic sodium dodecyl sulfate (SDS) in the absence of and presence of additives like urea and acetamide. The effect of additives on the cloud point (CP) of nonionic surfactants has been studied for more than six decades. Ionic surfactants, however, generally do not show clouding. But we recently, CP in ionic surfactant like anionic SDS have been reported in absence and presence of additives.

In most of their applications, surfactants with additives, rather than pure are preferred, as such systems often exhibit enhanced properties through synergism [10]. The ingredients commonly present in industrial products strongly affect their clouding behavior [11]. Therefore it is important to understand the magnitude and nature of these additives effects as well as the mechanism involved so that systems can be tailored to exhibit clouding behavior at desired temperatures. The effects of different additives on the CP behavior of nonionic surfactants have been investigated [9].Unlike that with nonionic's [12], CP variation in ionic surfactants as a function of the additives present currently lacks predictive power; therefore, it is important to establish a good CP database of systems undergoing clouding for their desired mode of applications [5]. An understanding of the intermolecular forces that give rise to the clouding phenomenon observed experimentally is important for both application oriented reasons and as a means to test theories of phase transition and critical phenomenon [13]. Such experiments can involve variations in temperature and or additive(s)

In this paper, we explore both these effects; such studies may find use in the extraction of thermally labile compounds. Our earlier results on SDS + Urea and SDS + Acetamide helped identity combinations for the present purpose to study the effect of additives. The chosen combinations have wider temperature windows for making variations below and above their CP. The effects of additives on the CP of sodium dodecyl sulfate (SDS) were reported in this study.

## EXPERIMENTAL SECTION

Anionic surfactant, Sodium Dodecyl Sulfate (SDS) Mol. Wt. = 288.372 g/mol was obtained from Loba Chemie, India and it was used as received. The additives urea and acetamide was the product of Sigma Aldrich, USA (Urea Mol. Wt. 60.60 and Acetamide Mol. Wt. 59.07). Both the additives are dialyzed to remove low molecular weight fractions and other associated electrolytic impurities before use.

Deionized doubly distilled water having a specific conductivity in the range 1-2 x $10^{-6}$ S $cm^{-1}$ at 303.15 K was used in the preparation of all solutions of different concentrations. The CP was measured by visual observations of the abrupt change in the appearance of the surfactant solution from being clear to the first turbidity [14].

Pure sodium dodecyl sulfate solutions of different concentrations and containing the mixed additives were equilibrated at room temperature for one hour before measuring the cloud point temperature. The cloud point of a micellar solution of pure SDS surfactant and with mixed system was determined by visual observation of the temperature at which the clear solution turns turbid upon being heated up and vice versus on cooling. The samples were heated at a rate of 1ºC $min^{-1}$ the temperature exceeded the CP, solution was cooled below the CP temperature and then in was heated again to check the reproducibility of the measurement. The maximum uncertainty in the CP measurement was ± 0.5ºC.

2.2. Clouding Species
2.2.1 Surfactant- Anionic Surfactant
Sodium Dodecyl Sulfate (SDS) Mol. Wt. 288.372 g/mol (Loba Chemie)
Molecular formula-$CH_3(CH_2)_{11}OSO_3Na$

2.2.2. Additives -
a) Urea: - Mol. Wt. 60.60 g/mol.
Molcular formula-$CH_4N_2O$ $NH_2 – CO – NH_2$

b) Acetamide:- Mol. Wt. 59.07 g/mol
Molcular formula-$C_2H_5NO$ $CH_3$-CO – $NH_2$

## RESULTS AND DISCUSSION

3.1. Cloud points (CP) of Sodium Dodecyl Sulfate (SDS)
Cloud points of SDS in aqueous media yet not reported but we recorded CP of SDS in acidic medium which is able to form cloud point at ambient temperature, it requires concentrated hydrochloric acid to induce cloud point phenomenon and phase separation [15-17]. Thus in SDS the surfactant rich phase has high acidifies ($p^H < 2$) it requires much more dilution and pH adjustment in order to obtain cloud points. The cloud points of pure sodium dodecyl sulfate, anionic surfactant at different concentrations in weight percent (Wt %) (w/v) from 0.01to 0.1Wt % (CMC = 8.2 mM) are given in Table 1. The CP of pure SDS is substantially near most values over a wide range of concentration. The values of CP increase rapidly initially and then mildly from $14.1^0$C to $31.6^0$C with increase in concentration of sodium dodecyl sulfate surfactant from 0.01 to 0.1 Wt % An increase in CP of SDS with increasing concentration shows that there is stabilization of the interface between the hydrophobic core of the micelles of SDS and water which is considered to be a major driving force for water-SDS interaction [14].

**Table 1. CP of SDS (pure anionic surfactant) at different concentration weight percent**

| [SDS] Wt % | Molarity x $10^{-4}$ mol/dm$^3$ | Mol fraction $X_s$ x$10^{-5}$ | ln$X_s$ | CP $^0$C |
|---|---|---|---|---|
| 0.01 | 0.6230 | 0.6236 | -11.985 | 14.1 |
| 0.02 | 0.6935 | 1.2473 | -11.292 | 16.6 |
| 0.03 | 1.0400 | 1.8710 | -10.886 | 18.5 |
| 0.04 | 1.3870 | 2.4948 | -10.598 | 21.5 |
| 0.05 | 1.7338 | 3.1187 | -10.375 | 24.5 |
| 0.06 | 2.0800 | 3.742 | -10.193 | 26.9 |
| 0.07 | 2.4270 | 4.3659 | -10.039 | 28.4 |
| 0.08 | 2.7700 | 4.9896 | -9.905 | 29.6 |
| 0.09 | 3.1209 | 5.6132 | -9.787 | 30.4 |
| 0.1 | 3.4677 | 6.23694 | -9.682 | 31.6 |

3.2. Cloud points (CP) SDS-Urea as additive system

The influence of Urea on the cloud point of sodium dodecyl sulfate at different concentrations has been also studied. The results are given in Table 2. These results indicating that the cloud point of SDS surfactant declined considerable with decrease in concentrations of additives 0.05 to 0.005 Wt%. The CP decreases from 21$^0$C to 10.0$^0$C for 0.1 Wt% SDS with an increasing in the concentration of Urea from 0.005 to 0.05 Wt% which is in conformity with earlier findings [18-20]. This is due to availability of urea $H_2N^+$-CO-$NH_2^-$ more near the micellar surface, leading to increased hydrophobic interactions and micellar charge neutralization. As a consequence, removal of water from the head group region is facilitated, causing a decrease in CP. To generalize the effect, we considered the effect of added urea on 0.1to 0.001Wt% SDS solutions of different concentrations. The general trend observed is an increase in the CP with the increase in Urea concentrations (Figure 1, 2).

Urea and thiourea believed to be structure breakers and are found to increase the CP values of nonionic surfactants [21, 22]. Two different mechanisms for urea action on micellar solutions have been proposed: (i) Urea changes the structure of water to facilitate the solvation of a hydrocarbon chain; (ii) urea replaces several water molecules that solvate the hydrophobic chain and the polar head group of amphiphilic. The first mechanism is widely accepted and many experimental results seem to support the hypothesis that urea acts as a water structure breaker [23].

In particular, the addition of urea to surfactant solutions leads to an increase in the CMC value. Jencks and coworkers have proposed that the increased solubility of hydrocarbons in aqueous urea results primarily from a smaller free energy of cavity formation in the mixed solvent, resulting in the replacement of water by a larger urea molecule in the salvation region [24, 25]. It has also been reported that the degree of contention dissociation (β) of micelles increases with urea addition [26]. As a result of the increase in β, the micelle hydration would increase, which would contribute to the CP increase. The two factors taken together seem to be responsible for the CP increase.

**Table 2. Influence of [Urea] on CP of SDS**

| [SDS] | CP $^o$C at [Urea] Wt % | | | | | |
|---|---|---|---|---|---|---|
| Wt % | 0.005 | 0.01 | 0.02 | 0.03 | 0.04 | 0.05 |
| 0.01 | 0.9 | 2.2 | 3.4 | 5.0 | 6.3 | 7.0 |
| 0.02 | 3.1 | 4.1 | 5.2 | 6.0 | 7.1 | 8.1 |
| 0.03 | 4.4 | 5.3 | 6.8 | 7.0 | 8.2 | 9.8 |
| 0.04 | 5.3 | 6.1 | 7.4 | 8.1 | 9.0 | 10.9 |
| 0.05 | 6.1 | 7.8 | 8.2 | 10.0 | 11.6 | 12.0 |
| 0.06 | 7.4 | 8.5 | 9.9 | 11.1 | 13.1 | 13.5 |
| 0.07 | 8.3 | 9.8 | 10.4 | 12.0 | 14.0 | 14.3 |
| 0.08 | 9.1 | 10.0 | 11.0 | 13.4 | 15.3 | 16.3 |
| 0.09 | 9.7 | 10.4 | 11.5 | 14.0 | 16.4 | 19.9 |
| 0.1 | 10.0 | 11.3 | 12.4 | 14.9 | 17.0 | 21.0 |

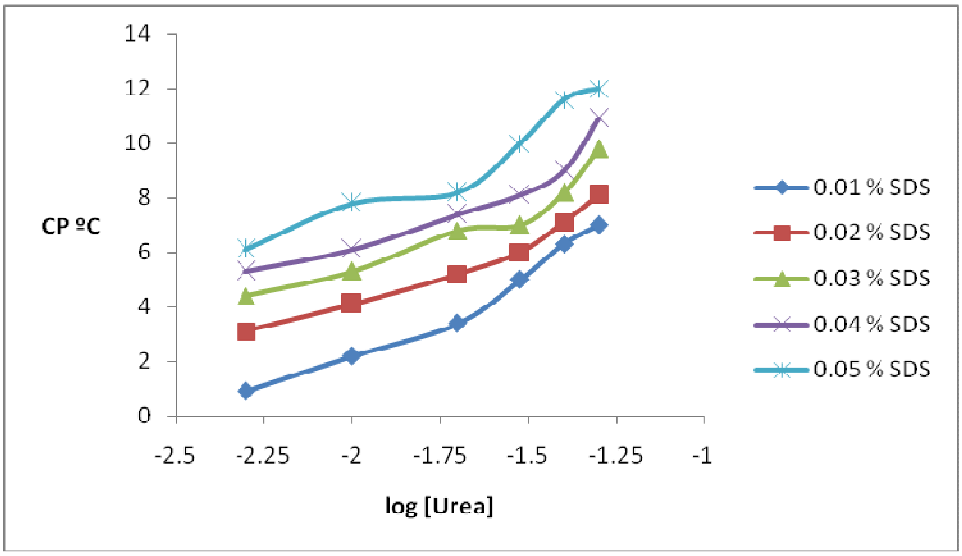

**Figure 1.Influence of [Urea] on CP of SDS at various concentration from 0.01to 0.05 weight percent**

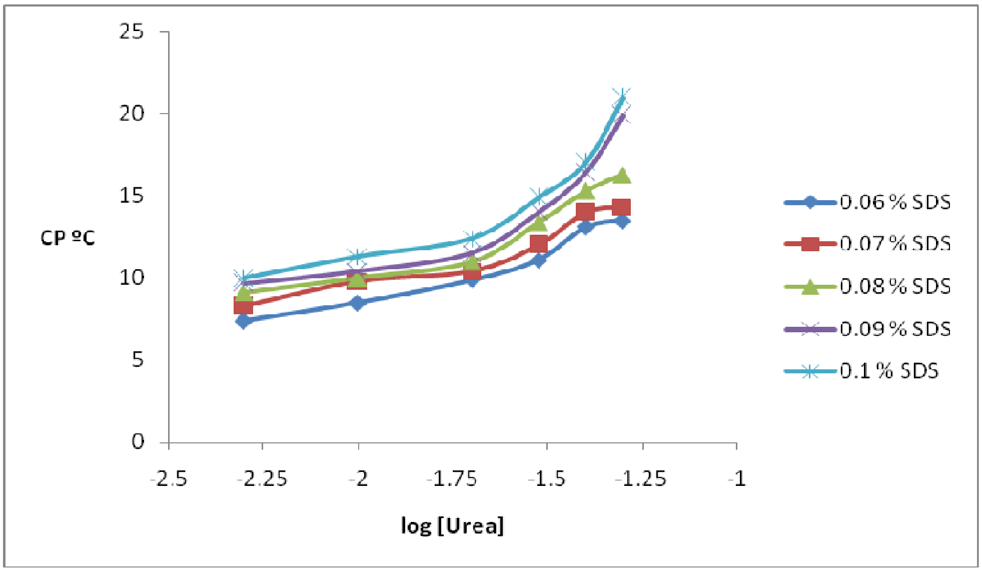

**Figure 2.Influence of [Urea] on CP of SDS at various concentrations from 0.06to 0.1 weight percent**

3.3. Cloud points (CP) SDS-Acetamide as additive system

The effect of addition of organic additives, acetamide to the on CP of SDS 0.1 to 0.001 Wt% concentrations was shown in Figure 2. The results are given in Table 3. This mixed system shows a decrease in CP with decreasing acetamide concentrations can be seen. Acetamide organic additive was tested to evaluate their effect on the CP of the SDS concentration systems in Table 3. This organic additive was found to decrease the CP, the rate of decrease of CP was the highest as compared Urea. Polar organic compound usually partition in the head group region with the alkyl chain penetrating into the micellar core. This partitioning results in replacement of the water from the micellar head group region; hence a lower temperature is required to show clouding. Hydrophilic ranking for a few polar organic compounds has been discussed [27]. Aliphatic hydrocarbons usually partition inside the micellar core ; hence urea is the least effective than acetamide in replacing water from the head group region and in turn in decreasing the CP. In the case non-ionic surfactants, the presence of aliphatic hydrocarbons raises the CP, whereas the other polar compounds have a decreasing effect by becoming solubilized in the poly oxy ethylene of the micelle [28]. Figure 3 and 4 illustrates the effect of acetamide on the organic additives the CP of the 0.1 to 0.001 Wt% SDS mixed system. Both additives (urea and acetamide) decrease the CP, but acetamide decreases the CP more rapidly. This can be understood from the discussion of the CP decreasing with addition of urea, where the presence of $H_2N$-CO-$NH_2$ group enables the micelle to interact specifically with the compound. A sharp decrease in CP is observed with acetamide, owing to the presence of an aliphatic moiety in acetamide that enables the hydrophobic interactions with the SDS micelle. Because amide group is also there, the additive seems to have a closer contact with the micellar surface replacing water from the head group region and resulting in a decrease in the CP.

**Table 3. Influence of [Acetamide] on CP of SDS**

| [SDS] Wt % | [Acetamide] CP °C Wt%<br>0.005 | 0.01 | 0.02 | 0.03 | 0.04 | 0.05 |
|---|---|---|---|---|---|---|
| 0.01 | 0.9 | 11.2 | 13.2 | 14.1 | 15.3 | 19.1 |
| 0.02 | 10.1 | 12.4 | 14.0 | 15.4 | 16.0 | 20.2 |
| 0.03 | 11.2 | 13.1 | 14.9 | 16.0 | 17.1 | 22.0 |
| 0.04 | 12.0 | 14.3 | 15.1 | 17.4 | 18.2 | 23.5 |
| 0.05 | 13.4 | 15.4 | 16.2 | 18.2 | 19.3 | 25.3 |
| 0.06 | 15.0 | 17.0 | 18.1 | 21.8 | 21.0 | 26.1 |
| 0.07 | 17.3 | 19.3 | 20.4 | 22.5 | 23.1 | 27.0 |
| 0.08 | 19.4 | 21.1 | 22.2 | 23.3 | 24.3 | 28.2 |
| 0.09 | 21.1 | 23.3 | 24.1 | 25.0 | 26.0 | 29.5 |
| 0.1 | 22.0 | 27.3 | 28.0 | 28.5 | 29.1 | 31.2 |

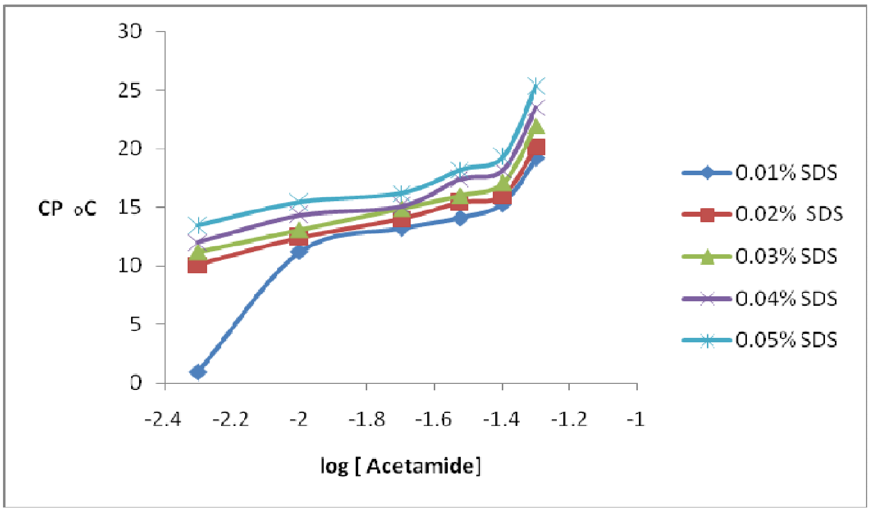

**Figure 3. Influence of [Acetamide] on CP of SDS at various concentration from 0.01to 0.05 weight percent**

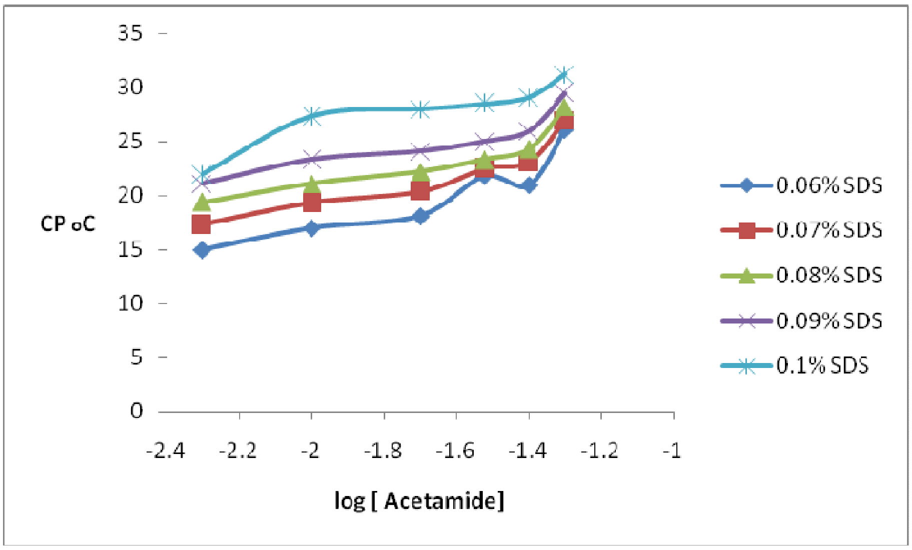

**Figure 4. Influence of [Acetamide] on CP of SDS at various concentrations from 0.06 to 0.1 weight percent**

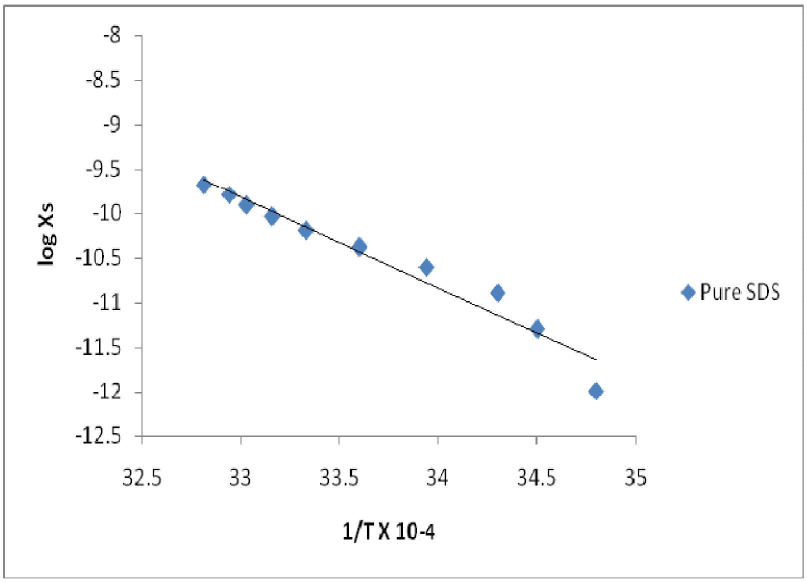

**Figure 5. log Xs Vs 1/T X $10^{-4}$ for pure SDS**

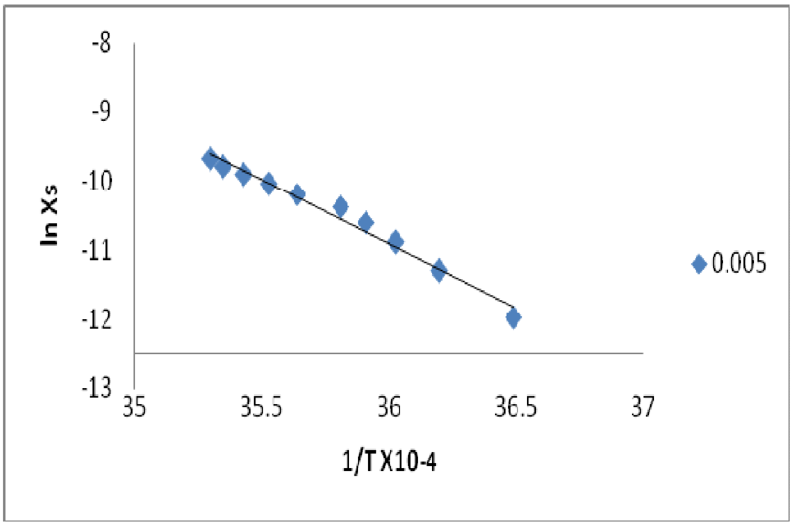

**Figure 6. log Xs Vs 1/T X $10^{-4}$ for SDS + 0.005 Urea**

3.4. Thermodynamics of Clouding

All physicochemical processes are energetically controlled. The spontaneous formations of micelle are obviously guided by thermodynamic principles. Cloud points are the characteristics of non-ionic surfactants, but, we studied the characteristics of anionic surfactant SDS with respective to cloud points. Thermodynamic parameters of solublization of pure sodium dodecyl sulfate are given in Table 4 and SDS-Urea, SDS-Acetamide mixed systems are given in Table 5 and 6 respectively.

Considering cloud point the phase separation point, the thermodynamic parameters such as standard free energy ($\Delta G^0_{cl}$), enthalpy ($\Delta H^0_{cl}$) and entropy ($\Delta S^0_{cl}$) for the clouding process have been calculated using the "Phase Separation Model" The following relation can be written as –

$$\Delta G^0{}_{cl} = RT \ln X_s \qquad \text{……(1)}$$

Where "cl" stands for clouding process and $\ln X_s$ is the mol fraction solubility of the solute. The standard enthalpy ($\Delta H^0_{cl}$) for the clouding process have been calculated from the slope of the linear plot of $\ln X_s$ Vs1/T in figure 5 for pure SDS and Figure 6 for SDS+0.005Wt% urea.

$$d\ln X_s / dT = \Delta H^0{}_{cl} / RT^2 \qquad \text{……(2)}$$

The standard free energy of the clouding process$\Delta$ $S^0_{cl}$ have been calculated from the following relationship

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$$\Delta S^0_{cl} = (\Delta H^0_{cl} - \Delta G^0_{cl}) / T \qquad \text{……(3)}$$

The thermodynamic parameters for pure surfactant and in mixed systems are given in Table 4 and Table 5, 6 respectively.

$\Delta H^0_{cl} < \Delta G^0_{cl}$ indicating that overall clouding process is exothermic and also $\Delta H^0_{cl} > T\Delta S_{cl}$ indicate that the process of clouding is guided by both enthalpy and entropy. It has been observed that concentration of sodium dodecyl sulfate increases the$\Delta G^0_{cl}$ which increases from 24.53 to 28.62 kJ mol$^{-1}$ and $\Delta S^0_{cl}$ decreases -0.200 to -0.227 kJ mol$^{-1}$K$^{-1}$ respectively.

The present work would be supportive evidence regarding the probable interaction between anionic surfactants and additive molecules, organic compounds leading to the phase separation at the cloud point. The effect of urea and acetamide on the cloud point is a clear indication that the phenomenon of clouding is associated with the different micelles coalescing

**Table 4. Thermodynamic parameters of solubilization of pure SDS**

| **[SDS] Wt %** | **$\Delta G^0_{cl}$ kJ mol$^{-1}$** | **$-\Delta H^0_{cl}$ kJ mol$^{-1}$** | **$-\Delta S^0_{cl}$ J mol$^{-1}$K$^{-1}$** |
|---|---|---|---|
| 0.01 | 28.62 | 36.59 | 227.0 |
| 0.02 | 27.20 | | 220.0 |
| 0.03 | 26.40 | | 215.97 |
| 0.04 | 25.96 | | 212.28 |
| 0.05 | 25.68 | | 209.20 |
| 0.06 | 25.43 | | 206.69 |
| 0.07 | 25.17 | | 204.80 |
| 0.08 | 24.93 | | 203.20 |
| 0.09 | 24.70 | | 201.90 |
| 0.1 | 24.53 | | 200.55 |

**Table 5. Thermodynamic parameters of SDS in presence of Urea**

| **[Urea] Wt %** | **$\Delta G^0_{cl}$ kJ mol$^{-1}$** | **$-\Delta H^0_{cl}$ kJ mol$^{-1}$** | **$-\Delta S^0_{cl}$ J mol$^{-1}$K$^{-1}$** |
|---|---|---|---|
| 0.005 | 26.17 | 66.871 | 328.592 |
| 0.01 | 24.65 | 69.395 | 330.620 |
| 0.02 | 23.09 | 69.394 | 323.926 |
| 0.03 | 22.33 | 57.376 | 276.730 |
| 0.04 | 21.80 | 54.852 | 264.183 |
| 0.05 | 21.56 | 42.552 | 217.950 |

**Table 6. Thermodynamic parameters of SDS in presence of Acetamide**

| **[Acetamide] Wt %** | **$\Delta G^0_{cl}$ kJ mol$^{-1}$** | **$-\Delta H^0_{cl}$ kJ mol$^{-1}$** | **$-\Delta S^0_{cl}$ J mol$^{-1}$K$^{-1}$** |
|---|---|---|---|
| 0.005 | 27.21 | 33.43 | 205.451 |
| 0.01 | 25.97 | 38.05 | 213.080 |
| 0.02 | 24.30 | 39.60 | 212.187 |
| 0.03 | 23.32 | 44.40 | 224.499 |
| 0.04 | 22.61 | 41.98 | 213.714 |
| 0.05 | 22.24 | 62.34 | 277.923 |

## CONCLUSION

The effect of additives like urea and acetamide on CP of SDS were studied here, on the critical exponents constitutes an interesting problem. The effect of surface active additives the cloud point is a clear indication that the phenomenon of clouding is associated with the different micelles coalescing. The present paper supports the conjecture that the cloud point is a critical phenomenon. Our study of micellization behavior of anionic surfactant SDS in absence and presence of additives indicates that hydrophobic chains of polar compounds penetrate the micelle interior while the polar groups remain on the micelle surface, thereby reducing the amount of water near the head-group region. This results in appreciable lowering of CP due to dehydration of surfactant head groups, thus facilitating micellar growth. The CPs of the mixed systems SDS + Urea and SDS + Acetamide are significantly lower than that of the single pure SDS system due to presence of neutral or no charge in the micelles of the mixed system. At low concentrations both urea and acetamide, additives exert their effect on SDS that leads to dehydration of micelles and hence, a lowering of the CP. The process of clouding is associated with large changes in $\Delta H^0_{cl}$, $\Delta S^0_{cl}$ as well as$\Delta G^0_{c1}$ the entropy change suggests overall ordering of the system. It has been observed that concentration of sodium dodecyl sulfate increases the$\Delta G^0_{cl}$ which increases from 24.53 to 28.62 kJ mol$^{-1}$ and$\Delta S^0_{cl}$ decreases -0.200

to -0.227 kJ $mol^{-1}K^{-1}$ respectively. In case of SDS + Urea or Acetamide mixed systems$\Delta G^0_{cl}$ decreases and $\Delta H^0_{cl,}$ $\Delta S^0_{cl}$ increases respectively as concentration of additives increases from 0.005 to 0.05 Wt%. As $\Delta G^0_{cl}$ is positive the clouding process is non-spontaneous and $\Delta H^0_{cl}$ is negative clouding process in exothermic.

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