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Conductometric Approach to the Thermodynamic of Micellization of Anionic Surfactants in the Presence of Procaine Hydrochloride

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ABSTRACT

This study underscores the teleology of micellization behaviours of sodium dodecyl sulphate (SDS) and sodium lauroyl sarcosinate (SLS), an anionic surfactant. The configuration of SDS/SLS water soluble in the presence of 4.0 x 10^{-5} moldm^{-3} aqueous solution of procaine hydrochloride within the temperature range of 293.15 K to 313.15 K was observed. Critical micelle concentration (CMC), and degree of counter ion binding (β) were determined from conductivity data. The CMC of SDS and SLS decreased to reach a minimum (T= 308.15 K and 303.15 K for SDS and SLS) and then increased with increasing temperature. On applying mass action model, the obtained CMC values were used to determine the thermodynamic parameters (i.e. free energy of micellization (ΔG°_m), enthalpy of micellization (ΔH°_m) and entropy of micellization (ΔS°_m)). As a function of temperature, the ΔG°_m value was negative and the negativity was enhanced in surfactants-PHC medium as compared with aqueous medium. This is an indication that spontaneity increases in micelle formation in the SDS+PHC and SLS+PHC systems than water. Entropy-enthalpy compensation were observed on applying Lumry-Rajender-entropy compensation model. The observed compensation temperatures, T_c, for SDS and SLS, were not the same with and without PHC. SDS had T_c values of 302.8 ± 3.14 and 307.7±1.63, while for SLS, T_c = 305.46 ±3.14 and 307.33±2.18. A clear indication of enthalpy–entropy compensation phenomenon was observed.

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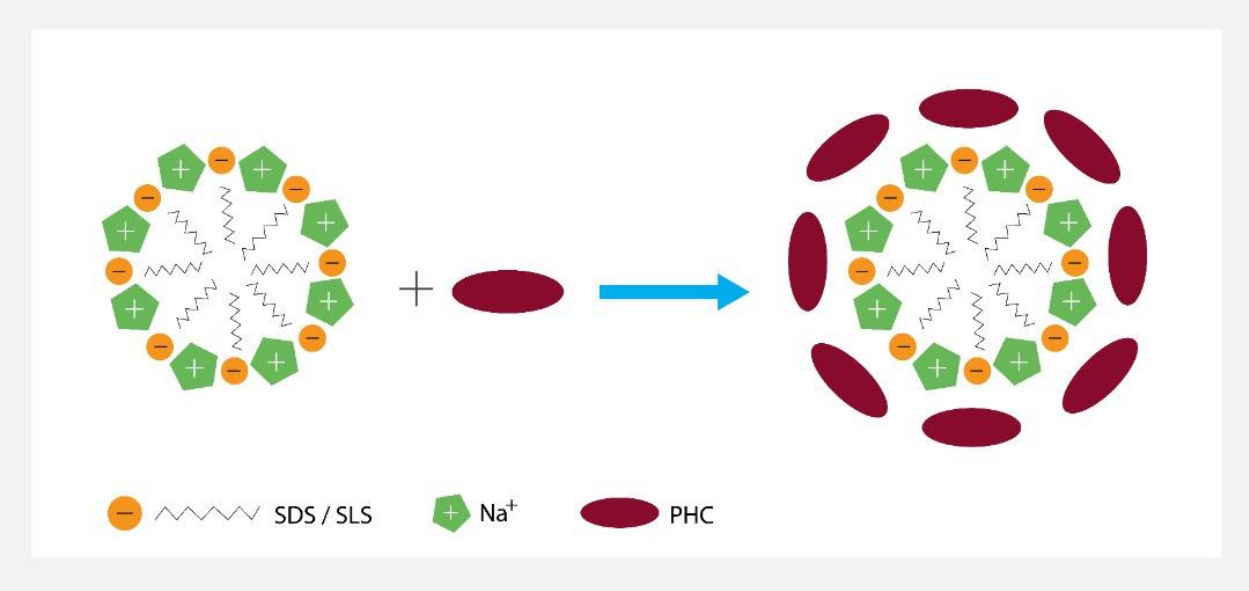
GRAPHICAL ABSTRACT



Introduction

Drugs are amphiphilic compounds, which consist of polar (hydrophilic) and non-polar (hydrophobic) functional groups. This invariably dictates its therapeutic activities and ability to interact with surfactants [1–4]. Activeness of any drug can be moderated based on the kind of interactions they experience in solution. Surfactants are also amphiphilic molecules which encapsulate polar (hydrophilic) and non-polar (hydrophobic) functional groups just like drug. As an amphiphilic compound, drug or surfactant aggregate at interface (i.e. micellized) [5–6]. The micellization or association phenomenon is caused by a precise balance of repulsive and attractive forces in the solution [1]. Procaine (Figure 1) and its derivatives are local anesthetic drugs, which are amphiphilic in nature alongside its colloidal properties [8]. Because of their closeness to the structure of natural molecules, they are employed to transmit nerve impulses [8]. The cationic form of the drug, which is the active principle, is thought to interact with the Na^+ channels on the neuron membrane, preventing nerve impulse initiation and transmission [9–11]. Anionic surfactants are used as excipients in pharmaceutical industries [7]. Little is known about its interaction with procaine hydrochloride (PHC), despite the immense biological and industrial applications, using conductometric technique [8]. It is imperative to say that drugs induce changes in physicochemical properties of aqueous surfactants solution, e.g. phase behavior and thermodynamic parameters, [12] and it is desirable to decipher the physicochemical properties of drugs, in relation to surfactants in solution as well as at the interface. Although, the interactions between PHC and biological membrane molecules, vis-à-vis their interactions with surfactant aggregate using different techniques have been reported [13–16], more importantly, there has been minimal focus on determining the impacts of PHC on the critical micelle concentration (CMC) and thermodynamic parameters of aqueous phase surfactant micellization. Because the presence of additives in an amphiphile affects its physicochemical properties (e.g. the degree of ionization, reaction

rates, CMC, and thermodynamic parameters) [17–22], we herein studied the thermodynamic of micellization of sodium dodecylsulfate (SDS), and Sodium lauroyl sarcosinate (SLS) (Figure 1) in the presence of local anesthetic drug (PHC), because they are regular ingredient used in industry.

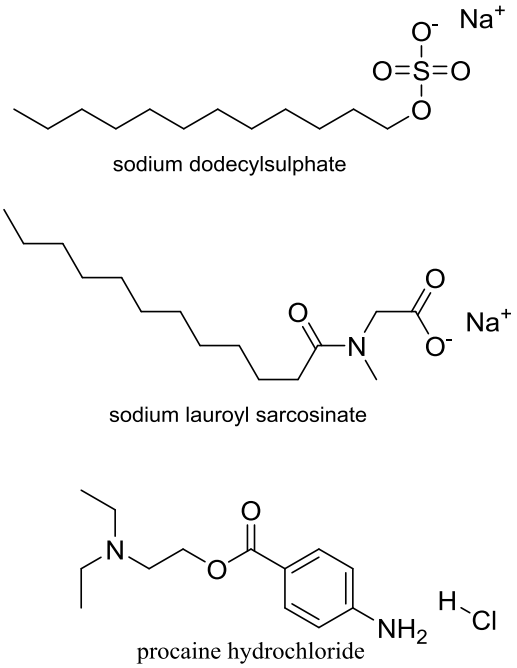


Figure 1. Molecular structure of sodium dodecyl sulfate (SDS), sodium N-lauroyl sarcosinate (SLS) and procaine hydrochloride (PHC)

Experimental

Materials

Deionized distilled water with an electrical conductivity of (1–2) $10^{-6} S.cm^{-1}$ and a pH of 6.8–7.0 (at T= 298.15 K) was prepared and utilized in all tests. Sodium dodecyl sulfate (SDS) (BioUltra ≥99.0%) and Sodium N-lauroyl sarcosinate (SLS) (≥ 98.0 %) were purchased from Sigma Aldrich, USA, and Procaine hydrochloride (PHC) (≥98.0%) from AKSci USA were of A.R. grade and were subsequently used without further purification.

Methods

A stock solution of 4.0 x10^{-5} mol/dm^3 PHC in pure water was made and utilized as the surfactant solution's solvent. To cover the pre- and post-micellar concentration ranges, surfactant solutions in aqueous were produced in concentrations ranging from 0.00041 to 0.01197 mol/dm^3 for SDS and 0.00073 to 0.02132 mol/dm^3 for SLS. All the solutions were prepared afresh for each experiment. A digital conductivity meter was used to test the electrical conductivities of surfactants in pure water and aqueous PHC. (Hanna-H15521-02). The conductivity meter was calibrated before use by measuring the electrical conductivities of 0.01 and 0.1 N potassium chloride solutions (Merck, purity ≥ 99%). In a thermostated beaker, a known volume of surfactants was titrated with a set amount of water (in the absence of an addition) or in the presence of an assumed concentration of PHC (4.0 x 10^{-5} M). In order to observe the effects of PHC on the micellization of SDS and SLS, solutions of both surfactants were prepared in aqueous solutions of PHC with

similar concentration. All measurements were made within 298.15 and 318.15 K in a thermostated water bath (Haake D8), maintaining the temperature constant within ±0.1 K. When the solution reached thermal equilibrium, the electrical conductivity was measured. The measurements were conducted three times and the average results were used in all computations. The electrical conductivity test has an accuracy of up to 1.3%.

Results and Discussion

Determination of Critical Micelle Concentration (CMC) and Degree of Counter-ion of Binding (β)

The CMC values of the two surfactants in the absence and presence of PHC in aqueous solution were used to analyze the interactions of SDS or SLS with PHC. At the break point in the particular conductivity-surfactant concentration graph, the CMC was calculated at T = (298.15, 303.15, 308.15, 313.15, 318.15) K. The conductivity raw data were fitted to the non-linear integral form of the Boltzmann-type sigmoid equation, which is a feature of the first derivative of the conductivity-concentration plot, as proposed by Carpena and others, to prevent inaccuracy in CMC estimation [23]. The Boltzmann sigmoid can be expressed as:

$$\frac{dk(c)}{dc} = \frac{A_1 - A_2}{1 + e^{(c-c_0)/\Delta c}} + A_2 \quad (1)$$

Where A_1 and A_2 denote the asymptotic values for small and large values of surfactant concentration c, respectively, c_o denotes the transition's center, and Δc denotes the transition's breadth. Integration of equation 1 yields:

$$k(c) = k(c=0) + A_1 c + \Delta c(A_2 - A_1)\ln\left[\frac{1 + e^{(c-c_0)/\Delta c}}{1 + e^{-(c_{0/\Delta c})}}\right] \quad (2)$$

Figure 2 depict a typical plot of specific conductivity against surfactant concentration using both differential conductivity and Carpena's approach. Based on Table 1, the observed CMC of SDS and SLS in water at various temperatures were consistent with the published values [24–27].

In the presence of PHC, the CMC of the surfactants is lower than in the absence (Table 1). The lower CMCs of both surfactants in aqueous PHC compared to pure water can be explained by two factors: (i) PHC solubilization between surfactant molecules in the outer portion of the micellar core, and (ii) ion-pair formation between PHC and surfactant molecules in the outer portion of the micellar core, or in the palisade layer of the micelles [28]. The reciprocal repulsion between the ionic head groups, as well as the effort required for micellization, diminishes as a result of solubilization, resulting in a drop in the CMC values of the surfactants in the presence of PHC. This observation has been reported, i.e. solubilization of additive on micellization, by many researchers [29–31]. Secondly, the strong electrostatic interaction between the negative groups ($-COO^-$ and $-SO_3^{2-}$) of micelles in the post-micellar region and the positive group $-\overset{+}{N}(CH_2)_2(CH_3)_2$ of PHC [30, 31] form an ion-pair. Once ion-pairs was formed, they assumed non-ionic surfactant characteristic, which increases hydrophobicity and large head groups [28, 30, 31]. This resulted in strong hydrophobic–hydrophobic interaction between the non-polar groups of the surfactants hence decrease in CMC of SDS and SLS value [28]. The possible location of PHC drug molecules in the SDS/SLS micelle is shown in scheme 1. Unexpectedly, the CMC decreased as temperature increased from 298.15K and dropped to minimum at 308.15K, which later began to rise (Table 1). However, the effect of temperature on CMC was governed by two opposing factors operating simultaneously: First, a reduction in hydrophobic hydration encourages micellization at low temperatures, and secondly, a decline in the hydrophilic hydration disfavored

micellization [32]. On account of partial dehydration of polar head groups at varied temperature owing to these two causes, an increase in repulsion appeared on polar head groups of both surfactant monomers in the bulk and the interfacial area of surfactant micelles. [33]. The first factor predominated over the second one at low temperature (i.e. between 298.15K to 308.15K) for both surfactants, leading to enhanced micellization, while the second factor predominated above 308.15K for both surfactant where micellization was not favored. This has demonstrated that when temperature rises, not only does the hydration of the hydrophilic group decrease, encouraging micellization, but it also causes the rupture of the structured iceberg around the hydrophobic surfactants chain, preventing micellization [33].

The degree of counter-ion of binding (β) were obtained from $(1-A_2/A_1)$ where $A_2/A_1=\alpha$. Using the α values, the degree of counter-ion of binding, β, is calculated as $\beta=1-\alpha$ and is given in Table 1. As shown in Table 1, it is obvious that for SDS and SLS, the degree of ionization rises with increase in temperature. The alkyltrimethyl ammonium bromides and sodium dodecyl sulphate showed similar kind of behaviour [34, 35]. Increase in α value as a function of temperature could be attributed to increase in thermal energy [36, 37].

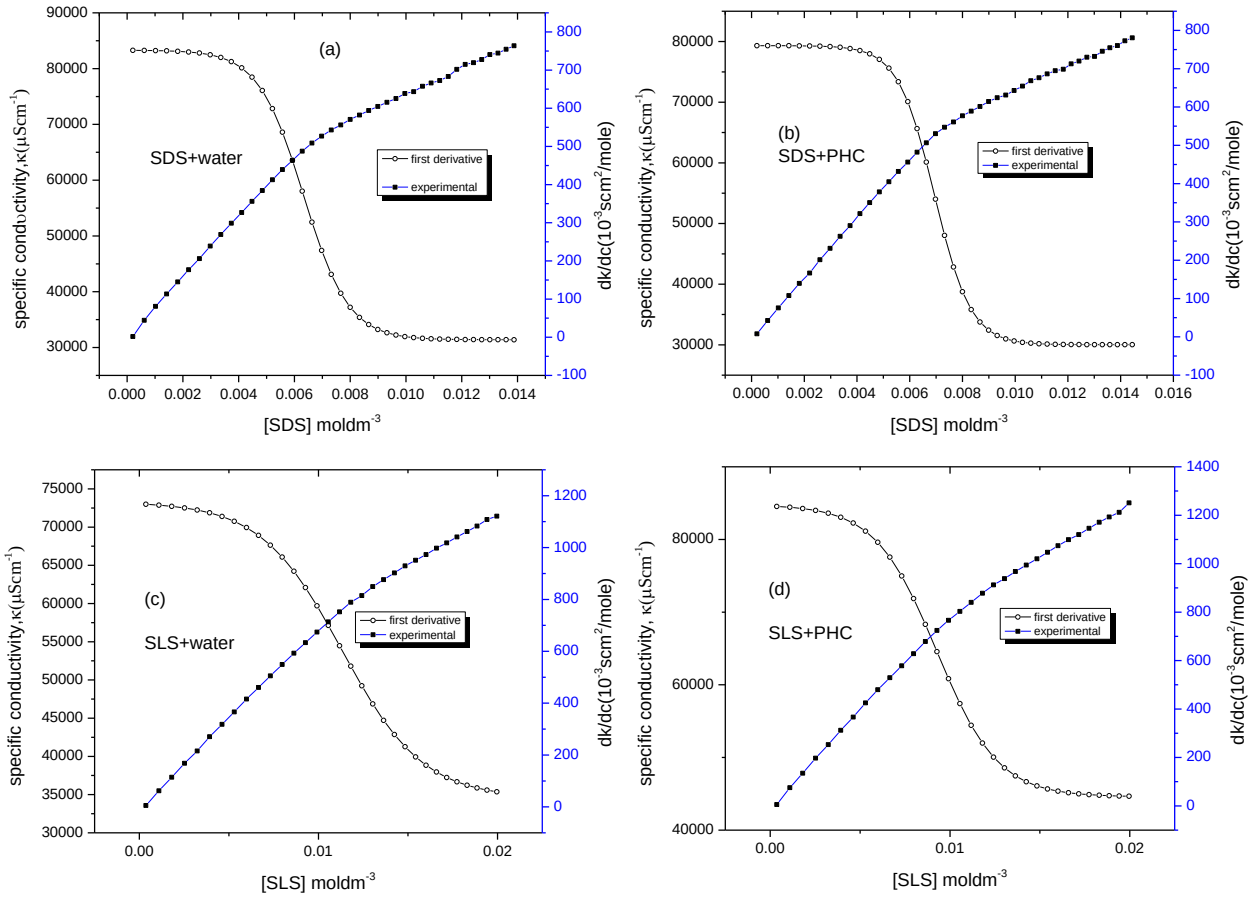


Figure 2. Variation of specific conductivity and differential conductivity with [SDS] (a) in water and (b) in 4.0 × 10^{-5} mol/dm³ PHC, with [SLS] (c) in water and (d) in 4.0 × 10^{-5} mol/dm³ aqueous PHC at different temperatures

Table 1. Variation of critical micelle concentration(CMC) and counter-ion binding (β) in the absence and presence of 4.0 x10^{-5} moldm^{-3} of PHC at different temperature

Surfactant	T(K)	CMC (mM)	β	$\beta_{average}$
SDS	298.15	8.32	0.58	
	303.15	7.90	0.57	
	308.15	7.10	0.56	0.58
	313.15	8.50	0.55	
	318.15	8.90	0.53	
SDS+ PHC	298.15	6.94	0.38	
	303.15	5.94	0.36	0.36
	308.15	5.26	0.35	
	313.15	6.10	0.34	
	318.15	6.50	0.32	
SLS	298.15	11.96	0.46	
	303.15	11.00	0.44	0.40
	308.15	13.20	0.43	
	313.15	14.10	0.36	
	318.15	15.40	0.32	
SLS+ PHC	298.15	11.17	0.49	
	303.15	9.75	0.43	
	308.15	8.18	0.46	0.47
	313.15	9.91	0.48	
	318.15	10.26	0.48	

Thermodynamics of Micellization in the Absence and Presence of 4.0 x 10^{-5} moldm^{-3} PHC

Temperature dependence of micellization of SDS and SLS with and without PHC has been studied as a measure of determining thermodynamic parameters of micellization. A plot of $\ln \chi_{cmc}$ against temperature for both systems PHC shows a minimum (Figure 3). In the absence and presence of PHC, the minimum occurs at 308.15K for SDS while for SLS it appears at 303.15K and 308.15K, respectively, which conform with the characteristic of an ionic surfactant with 12 carbon chain length [37, 38].

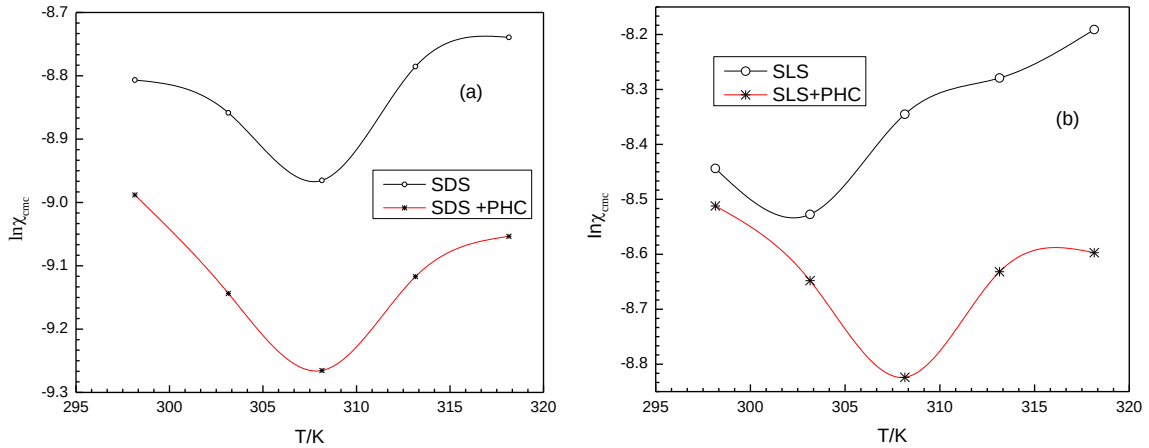


Figure 3. Plot of $\ln \chi_{cmc}$ *vs* temperature in aqueous and in 4.0 x 10^{-5} moldm^{-3} of PHC (a) SDS (b) SLS

Thermodynamic parameters such as, Gibbs free energy (ΔG_m^0), enthalpy (ΔH_m^0) and entropy(ΔS_m^0) needed for micellization of SDS and SLS with and without PHC were calculated by employing conductivity data on the basis of the phase separation model [39]. For ionic surfactants, the free energy of micellization ΔG_m^0 is given by the Equation [40, 41]:

$$\Delta G_m^0 = (2-\alpha)RT\ln(\chi_{cmc}) \quad (3)$$

Where χ_{cmc} is the value of CMC expressed on a mole fraction basis, defined as;

$$\chi_{cmc} = \frac{CMC}{CMC + [PHC] + 55.56} \quad (4)$$

The enthalpy of micellization ΔH_m^0 were obtained from the temperature dependence of the CMC using the Gibbs-Helmholtz Equation.

$$\Delta \mathrm{H}_m^0 = -RT^2(2-\alpha)[d(\ln \chi_{cmc})/dT]_p \quad (5)$$

The temperature dependence of the $\ln \chi_{cmc}$ was fitted to the equation derived by Kim and Lim for the temperature dependence of CMC [42];

$$\ln \chi_{cmc} = A_0 + A_1 \ln T + A_2/T \quad (6)$$

Where the A_0, A_1 and A_2 have been determined by a least square regression analysis. The fitting of the function in Eq. (4) to the variation of $\ln \chi_{cmc}$ with temperature for the micellization of SDS and SLS with and without 4.0 x 10^{-5} mol /dm^3 of PHC is shown in Figure 3. The entropy of micellization ΔS_m^0 were estimated from equation 3 and 5 respectively.
From equation 3 and 5,

$$\Delta S_m^0 = (\Delta \mathrm{H}_m^0 - \Delta G_m^0)/T \quad (7)$$

Thermodynamic parameters (i.e ΔG_m^0, ΔH_m^0 and ΔS_m^0) obtained from Equation 3, 5, and 7 have been summarized in Table 2. After further examination, it was discovered that the computed values for both systems in the absence and presence of PHC were negative, and that they became increasingly negative as the temperature rose, i.e. decreased with increasing temperature, but at a gradual rate. This is an evidence of the fact that micellization of these systems is thermodynamically favorable, spontaneous and enthalpy–entropy driven. The variation of ΔH_m^0 with temperature in absence and presence PHC is shown in Figure 4 for SDS and SLS. The ΔH_m^0 value is found to decrease linearly with increases in temperature; however, the data show that it is highly sensitive to temperature in all cases. As shown in Table 2, ΔH_m^0 > 0 below 303.15 K and ΔH_m^0 <0 above 303.15 K for both system in the absence of PHC. This behavior of ΔH_m^0 is in agreement with the proposal [44] that below 303.15 K, i.e., the temperature corresponding to the minimum in χ_{cmc}, micellization is entropy driven whereas above 303.15 K it is energy driven. In the presence of PHC, ΔH_m^0 > 0 below 308.15k and ΔH_m^0 <0 above 308.15k which is an evidence of interaction between SDS, SLS and PHC. For both systems, it is, however, of particular interest to note that increase in temperature caused both ΔS_m^0 and ΔH_m^0 values to decrease consistently, which agrees with the evidence available in literature [43]. As seen in Figure 4, ΔH_m^0 and ΔS_m^0 decrease as temperature rises, demonstrating that micellization is more energy driven at higher temperatures, and therefore compensates for enthalpy and entropy contributions, making ΔG_m^0 < 0 nearly temperature independent. It is expected that entropy change ΔS_m^0 be negative because micelle formation is a structure formation from surfactant monomers as obtained for SDS at 318.15k in the absence of PHC. Table 2 reveals

that the entropy change ΔS_m^0 is positive, indicating that surfactant aggregation is favored entropically. This positive number implies that iceberg clusters surrounding the surfactant monomer's hydrocarbon tails are melting, and the hydrocarbon chains in the micellar core are becoming more random [44]. At increasing temperatures, self-aggregation deteriorates an indication of decrease in ΔS_m^0 as indicated in Table 2. This is due to enhanced molecular motion at higher temperature [45].

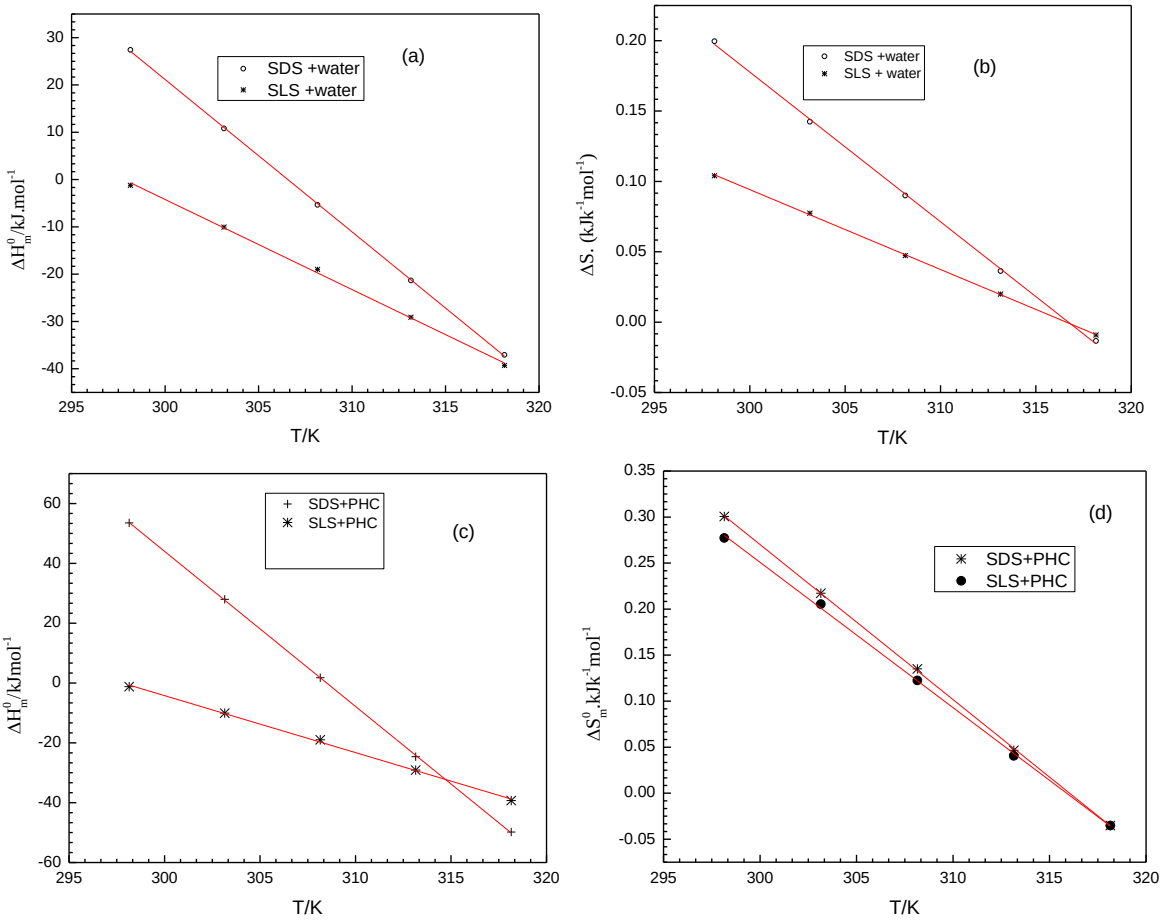


Figure 4. Plot of ΔH_m^0 and ΔS_m^0 *vs* temperature in absence and presence of 4.0 x 10^{-5} moldm^{-3} PHC

On account of Table 2, It is clearly shown that micellization process is both entropic and enthalpic driven with changes from entropic to enthalpic as temperature increases similarly, the micellization process loses entropic contribution, which is somewhat balanced by enthalpic gain. Figure 5 depicts the so-called enthalpy–entropy compensation effects, which are clearly defined by a linear relationship between enthalpy and entropy change. Similar behavior has been reported for different processes including micellization [46–48]. The correlation coefficient for this phenomenon, i.e., enthalpy-entropy compensation, is close to unity for the micellization of SDS and SLS with and without PHC. As proposed by Lumry-Rajender [46], the compensation phenomenon between ΔH_m^0 and ΔS_m^0 can be represented by the Equation;

$$\Delta H_m^0 = \Delta H^* + T_c \Delta S_m^0 \quad (8)$$

Table 2. Thermodynamic parameters for the micellization of SDS and SLS in the absence and presence of 4.0 x10^{-5}moldm^{-3} PHC

T/K	SDS + H_2O			SLS +H_2O		
	ΔG_m^0 (KJmol^{-1})	ΔH_m^0 (KJmol^{-1})	ΔS_m^0 (KJmol^{-1}k^{-1})	ΔG_m^0 (KJmol^{-1})	ΔH_m^0 (KJmol^{-1})	ΔS_m^0 (KJmol^{-1}k^{-1})
298.15	-32.09	27.41	0.20	-32.23	1.22	0.11
303.15	-32.37	10.21	0.14	-33.53	10.05	0.08
308.15	-33.07	-5.35	0.09	-33.57	-18.99	0.05
313.15	-32.71	-21.33	0.06	-33.35	-29.11	0.02
318.15	-32.83	-37.06	-0.13	-36.40	-39.30	0.01
	SDS +PHC			**SLS + PHC**		
298.15	-36.13	53.51	0.30	-31.79	50.88	0.28
303.15	-37.87	27.99	0.22	-34.28	27.99	0.21
308.15	-39.84	1.78	0.14	-34.92	2.83	0.12
313.15	-39.22	-24.64	0.05	-34.22	-21.52	0.04
318.15	-38.61	-49.79	0.04	-34.57	-45.75	0.03

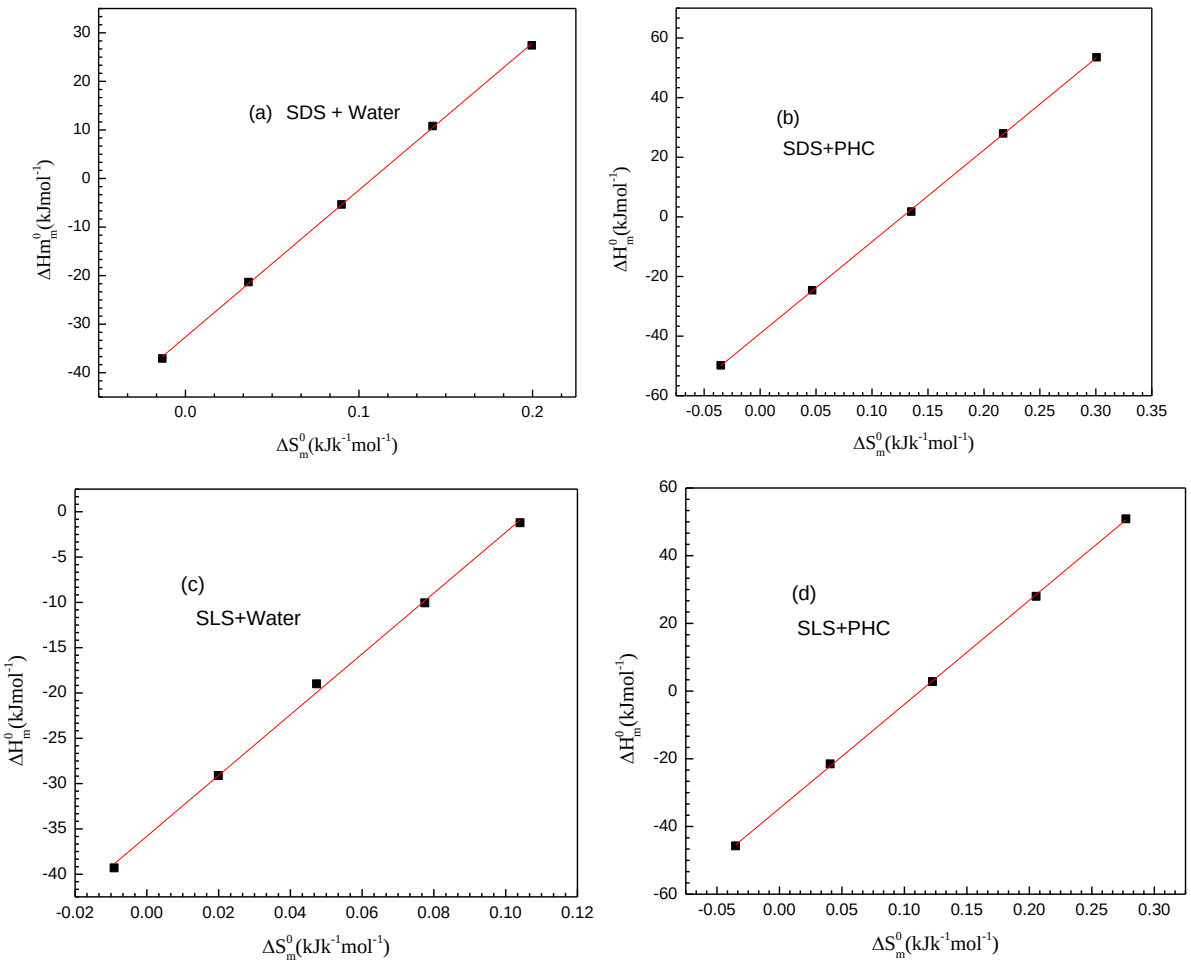


Figure 5. Plot of enthalpy-entropy compensation for the micellization of SDS/SLS with and without PHC

Where T_c (i.e the slope) in ΔH_m^0 versus ΔS_m^0 account for the compensation temperature and ΔH^* is the intercept of the enthalpy–entropy compensation plot. A typical plot is shown in Figure 5 below. In all, T_c is a measure of the de-solvation process of micellization. The ΔH^* is considers as the index of the chemical part of micellization (solute–solute interactions). It stands for the enthalpy effect in the absence of any entropic contributions (i.e. at $\Delta G_m^0 = 0$). For SDS and SLS in the absence and presence of PHC, the T_c values are not the same. SDS has T_c values of 302.8 ± 3.14 and 307.7 ±1.63 while for SLS, T_c = 305.46 ±3.14 and 307.33±2.18 respectively. This can best be interpreted that the organic additive PHC has significant effect on the de-solvation part of the micellization process.

The T_c values fits well to the general proposal made by Sugihara and Hisatomi, in which all surfactants should be included in the range from 299 to 315 K [48].

The values of H^* (i.e the intrinsic enthalpy gain) in the absence and presence of PHC are all negative indicating that the micellization process is favoured despite the fact that there is no entropic gain. The values are -32.66 ± 0.28, and -36.12 ± 0.29 for SDS and -34.67±0.36 and -35.81±0.45 for SLS respectively.

Conclusion

From the conductometric study, following conclusion were made:

- PHC had greater interaction with SDS than SLS, thereby, leaving an impact on both CMC and thermodynamics functions.
- The ΔG_m^0 value obtained was negative and the negativity was more enhanced in SDS+PHC / SLS+PHC system than in water, with increase in temperature.
- The negative correlation between the ΔG_m^0 and temperature increase showed that the micellization process is thermodynamically favorable and adequately spontaneous.
- micellization process was both entropic and enthalpic driven, with changes from entropic to enthalpic as temperature increases. This is an indication for enthalpy–entropy compensation phenomenon.

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Conflict of Interest

The authors declare that they have no competing interests.

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