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Thermodynamic Aspects of Micelle Formation of Ionic Surfactant–Polymer Interactions

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The thermodynamics of micellar aggregation involving ionic surfactants–sodium dodecyl sulfate (SDS) and sodium dodecylbenzene sulfonate (SDBS)–in the presence of guar gum (a natural polymer) and partially hydrolyzed polyacrylamide (PHPAM) have been systematically investigated using tensiometric and conductometric techniques. Critical micellar concentration (CMC) values for SDS and SDBS were accurately determined and the influence of polymer addition on these values was evaluated. Thermodynamic parameters, including the standard Gibb's free energy (ΔG^o_m), enthalpy (ΔH^o_m) and entropy (ΔS^o_m) changes of micellization, were calculated to elucidate the nature of the micellization process. The results provide significant insights into the physico-chemical interactions at the surfactant-polymer interface, offering potential implications for various industrial and environmental applications.

Keywords: Micellar aggregation, Critical micellar concentration, Surface tension, Surfactant–Polymer interactions.

INTRODUCTION

Surface-active agents (surfactants) are extensively utilized across a wide range of applications, including detergents, cosmetics, pharmaceuticals, enhanced oil recovery and environmental remediation such as oil removal from contaminated soils [1-3]. Surfactant mixtures have been shown to exhibit superior performance compared to individual surfactants in various industrial processes [4,5]. These mixtures are particularly beneficial in industrial formulations, pharmaceutical applications and oil recovery operations, where they enhance solubilization and interfacial activity [6].

The micellization of surfactants (amphiphilic molecules) occurs beyond a specific threshold concentration known as the critical micellar concentration (CMC), which is a key property in solution chemistry [7,8]. Determination of the CMC is essential for evaluating the thermodynamic parameters of the micellization process. These thermodynamic insights are crucial for understanding the spontaneity and stability of micelle formation, thereby enabling effective characterization and comparison of surfactant systems [9-12]. Surfactants reduce interfacial tension by migrating from one of two adjacent fluid phases to their shared interface, a process that releases potential energy and significantly alters the energy, entropy and dynamics of the system.

Several experiments have been conducted to investigate the interactions between two distinct anionic surfactants–sodium dodecyl sulfate (SDS) and sodium dodecylbenzene sulfonate (SDBS)–and two polymers, partially hydrolyzed polyacrylamide (PHPAM) and guar gum, in the presence of alkali (NaOH). These interactions were analyzed by measuring the surface tension and electrical conductivity of the solutions [13-25], but the complicated aggregation behaviour of surfactant-polymer mixtures with variations of temperatures are rarely investigated. Although earlier studies frequently reported the standard Gibbs free energy of micellization (ΔG^o_m), comprehensive thermodynamic analyses remain inadequate. Specifically, there is a lack of systematic comparative investigations involving other key thermodynamic parameters such as the standard enthalpy (ΔH^o_m) and standard entropy (ΔS^o_m) of micellization, particularly across varying temperatures [26-33]. Furthermore, significant gaps persist in the literature regarding the influence of different polymer concentrations on these thermodynamic parameters, which are essential for a deeper understanding of surfactant–polymer aggregation behaviour. In this study, a comparative thermodynamic analysis

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was conducted using two ionic surfactants and two polymers to evaluate the stability and molecular interactions of aggregated species across varying concentrations and temperatures. The selected surfactants are sodium dodecyl sulfate (SDS) and sodium dodecylbenzene sulfonate (SDBS), while the polymers include guar gum (a natural polymer) and partially hydrolyzed polyacrylamide (PHPAM). The CMCs of these surfactants were determined at different temperatures through surface tension and conductivity measurements, enabling a detailed investigation of micellization behaviour and thermodynamic properties [34,35]. The experimental results were analyzed to extract energetic information related to the self-aggregation of the amphiphiles and to evaluate the consistency between thermodynamic parameters obtained through pseudo-phase-model, van't Hoff method [36-38]. The findings are discussed with a pragmatic approach to elucidate the thermodynamic interactions governing the behaviour of surfactant–polymer systems.

EXPERIMENTAL

Sodium dodecyl sulfate (SDS, $C_{12}H_{25}NaO_4S$, m.w. = 288.38), (≥ 99% purity) is an anionic surfactant, purchased from Central Drug House (Pvt.) Ltd., India. Sodium dodecylbenzene sulfonate (SDBS, $C_{18}H_{29}NaO_3S$, m.w. = 348.48) (≥ 98% pure quality), guar gum (natural polymer) and partially hydrolyzed polyacrylamide (PHPAM) were purchased from LOBA Chemical Pvt Ltd., India for this study.

Preparation of solutions: The solutions containing varying concentrations of polymer and surfactant were freshly prepared before every experiment. Solutions were agitated using a Remi Magnetic Stirrer (REMI 2MLH) for roughly 15 min to prevent degradation. A broad range of concentrations that are close to the critical micellization values of SDS (0.01-0.4 wt.%) and SDBS (0.0001-0.4 wt.%) were used for the present investigation [30]. PHPAM and guar gum, two polymers, were employed at concentrations of 500 ppm to 5000 ppm [3,7]. Polymer concentration ranges (0.5-1.0 wt.%) were selected to match those for industrial applications [16,17].

Conductivity measurements: The conductivity data of the surfactant-polymer solutions were obtained by digital conductivity meter (Systronics, Model- 304).

Surface tension measurements: An Auto Tensiometer (model: 6801ES with platinum ring) was used to measure surface tension using the ring method at atmospheric pressure. The ring approach is predicated on the idea that a phase boundary area between liquid and air or liquid and liquid cannot be expanded until some resistance has been overcome. This resistance is directly measurable in mN/m with an accuracy of ±0.1 mN/m and is proportional to the surface and interfacial tension present. Prior to every measurement, the platinum ring was meticulously cleaned and flame dried. The vertically hung ring was dipped into the liquid and then removed to measure the surface tension. The surface tension, γ (mN/m), was determined to be the greatest force needed to pull the ring through the interface. At 298 K, distilled water was used to calibrate the tensiometer. Surface tension readings are presented as the mean values derived from a minimum of three replicate measurements. The double distilled water used to prepare the solutions had a surface tension of σ = 68 mN/m. Surface tension for a series of solutions were measured at different temperatures (298 K, 303 K, 313 K and 323 K) in presence and absence of polymers *e.g.*, guargum and partially hydrolyzed polyacrylamide (PHPAM).

RESULTS AND DISCUSSION

Tensiometric analysis: Tensiometric investigations of surfactant–polymer interactions reveal that as the concentration of surfactant (SDS or SDBS) increases, the surface tension of the solution initially decreases, reaches a minimum, then slightly increases and finally stabilizes. This initial decrease in surface tension is attributed to the adsorption of surface-active molecules at the air–solution interface. As the surface becomes saturated with surfactant molecules, micelle formation begins in the bulk solution. Beyond the CMC, further increases in surfactant concentration do not affect surface tension, as no additional adsorption occurs at the interface. Thus, the CMC can be accurately determined from the point at which surface tension ceases to decrease with increasing surfactant concentration.

The primary function of surfactants is to reduce the surface tension between an aqueous solution and another phase. However, in case of ionic surfactants, the presence of polymers in the aqueous medium significantly modifies these surface-active properties. When a polymer is introduced into a surfactant solution, the system becomes more complex, with various species coexisting in equilibrium. These include polymer-free surfactant aggregates, singly dispersed surfactant molecules, polymer-surfactant complexes and possibly free polymer chains. The formation of cross-molecular assemblies between the polymer and surfactant introduces additional interactions that influence micellization and interfacial behaviour.

SDBS exhibits a lower CMC (0.075 wt.%) compared to SDS (0.1 wt.%) due to its molecular structure. The hydrophobic tail of SDBS, which includes a bulky aromatic benzene ring, renders it more hydrophobic than the linear alkyl chain of SDS. This structural feature promotes more efficient packing within micelles. Furthermore, the sulfonate head group of SDBS generates lower electrostatic repulsion than the sulfate group in SDS, further contributing to micelle stability and reducing the CMC.

Guar gum exhibits a concentration-dependent effect on the surface tension of aqueous solutions of surfactant. At low surfactant concentrations, the surface tension is reduced, likely due to the combined surface activity of the polymer and the surfactant. As the concentration increases, the surface tension curve shows a breakpoint and then reaches a plateau, the extent of which is approximately proportional to the polymer concentration. Beyond this plateau, a further decrease in surface tension is observed, trending toward values similar to those of surfactant solutions without polymer.

The onset of surfactant–polymer interaction occurs at the critical aggregation concentration (CAC), identified as CAC1 and CAC2 for the SDS–guar gum and SDBS–guar gum systems, respectively (Fig. 1). At this point, the surface tension begins to decline again, indicating the formation of surfactant–polymer aggregates, although the increase in surface activity slows.

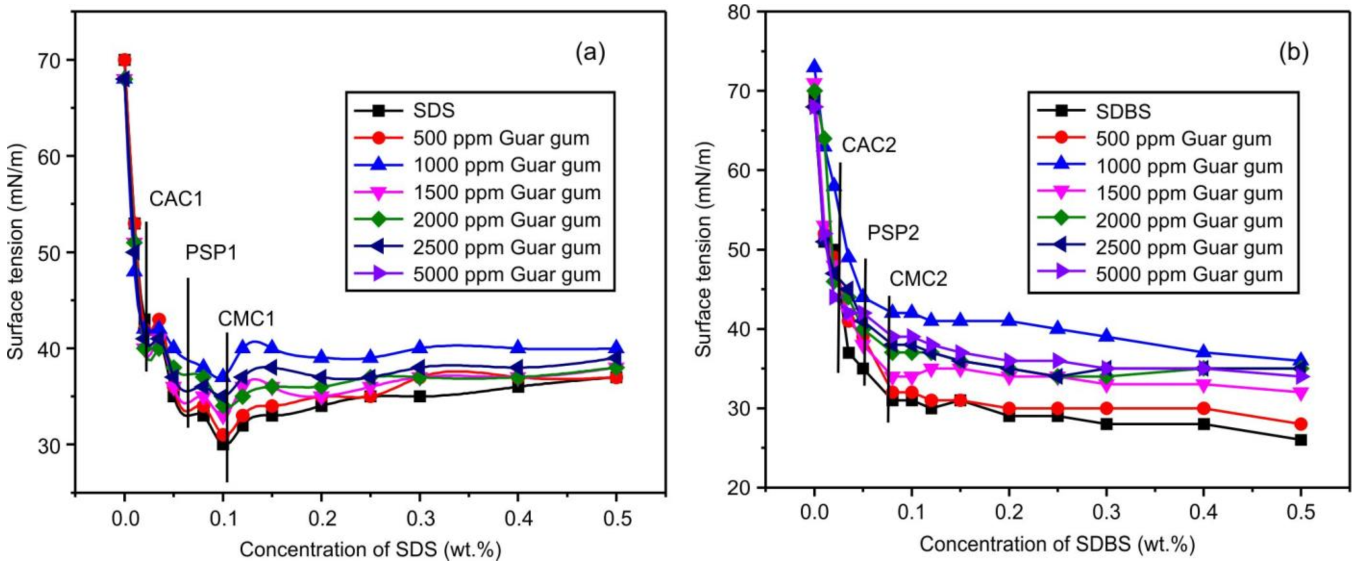

Fig. 1. Effect of guar gum on surface tension of (a) SDS and (b) SDBS at 298 K

Interestingly, the surface tension of the polymer–surfactant mixtures (guar gum with SDS or SDBS) was found to be higher than that of the corresponding surfactant solutions without polymer. This is attributed to the adsorption of guar gum onto the surfactant molecules through electrostatic and hydrophobic interactions, which hinders the free availability of surfactant molecules at the air–solution interface and reduces their surface activity.

In case of SDS, a slight increase in surface tension was observed upon further surfactant addition, reaching a maximum at around 1000 ppm (Fig. 1a), before decreasing again. This kind of surface tension maximum is uncommon in ionic the surfactant–ionic polymer systems, indicating a complex interaction mechanism. As the surfactant concentration increases above the CAC, interactions between the water-soluble polymer and surfactant intensify. Just above the polymer saturation point (PSP)–labeled PSP1 and PSP2–the system reaches a dynamic equilibrium, where both conventional micelles and surfactant-saturated polymer complexes coexist. Beyond the PSP, further addition of surfactant does not alter surface tension significantly, until the CMC is reached CMC1 and CMC2 for the SDS and SDBS–based systems, respectively (Fig. 2). At this point, the polymer becomes fully saturated with surfactants, typical micelle formation resumes, surface activity increases again and surface tension begins to decline and eventually stabilizes (Fig. 2).

The effect of partially hydrolyzed polyacrylamide (PHPAM) polymer on the surface tension of an aqueous solution of SDS and SDBS at 298 K is studied and analyzes through surface tension *vs*. concentration curve (Fig. 3). PHPAM formed a strong bond with surfactants through hydrophobic and hydrophilic interaction [39]. Both SDS and SDBS have negative charges so there is an electrostatic repulsion with negatively charged (carboxylate group) PHPAM that predominated by hydrophobic inter-action between bulky hydrophobic tail of SDBS and PHPAM. Thus, SDBS formed more stable polymer-

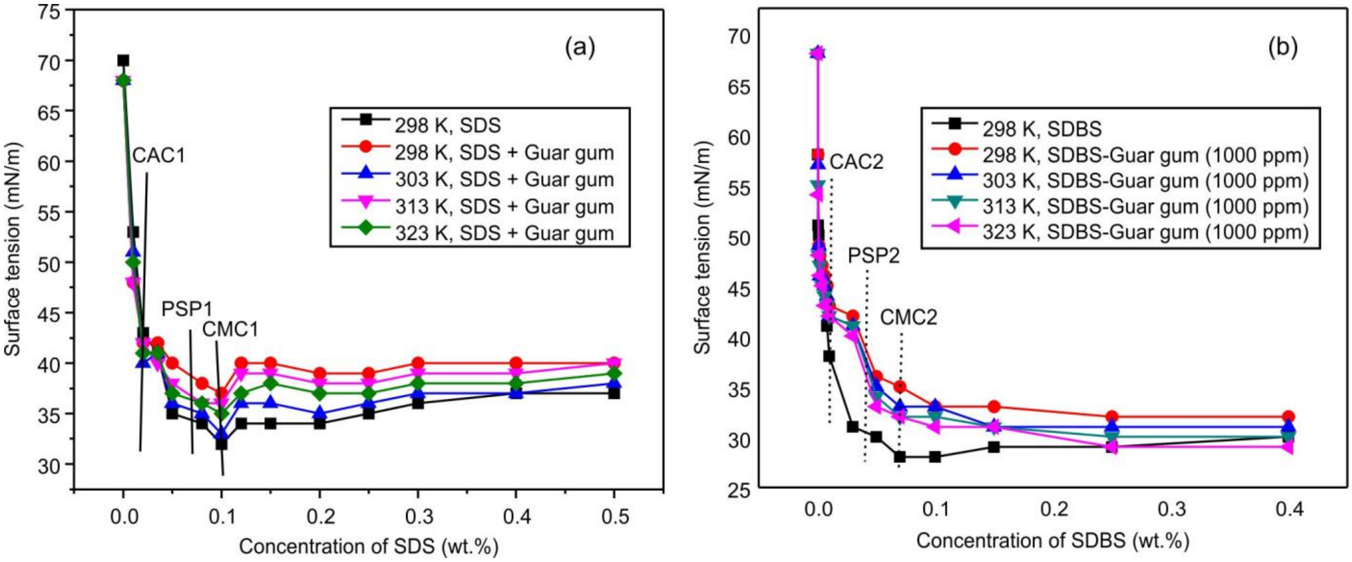

Fig. 2. Effect of guar gum (1000 ppm) on surface tension of (a) SDS and (b) SDBS at different temperatures

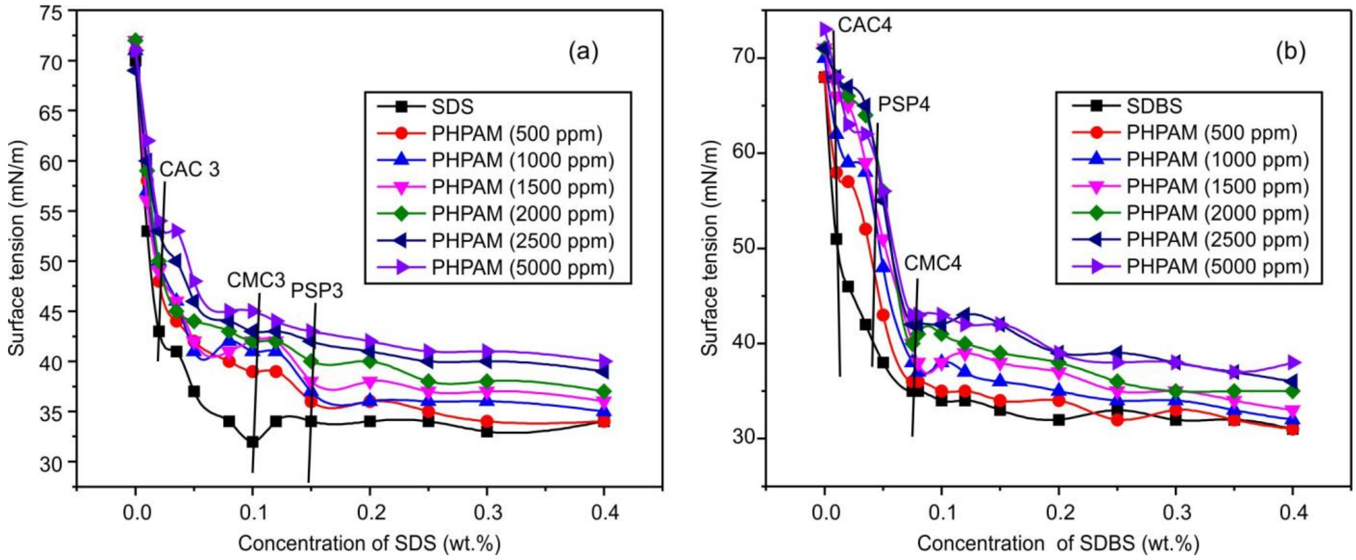

Fig. 3. Effect of PHPAM on surface tension of (a) SDS and (b) SDBS at 298 K

surfactant complexes with PHPAM than SDS-PHPAM interaction. The maximum concentration of PHPAM in SDS and SDBS solution is 1500 ppm that shown the maximum surface tension, after that the surface tension reduces due to the saturated polymer chain. The kinetic energy of molecules at the liquid-air interface increases with temperature, which reduces the cohesive forces of solution. As a result, the surface tension of solution decreases with rise in temperature. Surfactants (SDS and SDBS) with guar gum mixture show the minimum surface tension at 303 K (Fig. 2) and at higher temperature (more than 303 K) the surfactant mobility increases, which results in lower polymer-surfactant interaction (Fig. 4) [40].

Thermodynamics of micellization: The thermodynamic parameters of micellization were evaluated using the pseudo-phase model in conjunction with the van't Hoff approach. Surface tension measurements were carried out for SDS and SDBS solutions over a concentration range of 500 ppm to 5000 ppm at 298 K, both in the absence and presence of polymers such as guar gum and partially hydrolyzed polyacrylamide (PHPAM) [36].

The degree of micelle dissociation (α) and the corresponding counterion binding fraction [β = (1 – α)] were determined from the ratio of the slopes of surface tension *versus* logarithm of concentration plot above and below the CMC. These parameters are critical for quantifying the extent of counterion binding and the micellar charge environment.

The following equations were employed to calculate the thermodynamic parameters of micellization per mole of surfactant monomer:

$$\Delta G^{o}_{m} = 2RT(0.5+\beta)\ln X_{CMC} \tag{1}$$

$$\Delta H^{o}_{m} = -2RT^{2}(0.5+\beta)\left(\frac{\partial \ln X_{CMC}}{\partial T}\right)_{p} \tag{2}$$

$$\Delta S^{o}_{m} = \left(\frac{\Delta H^{o}_{m} - \Delta G^{o}_{m}}{T}\right) \tag{3}$$

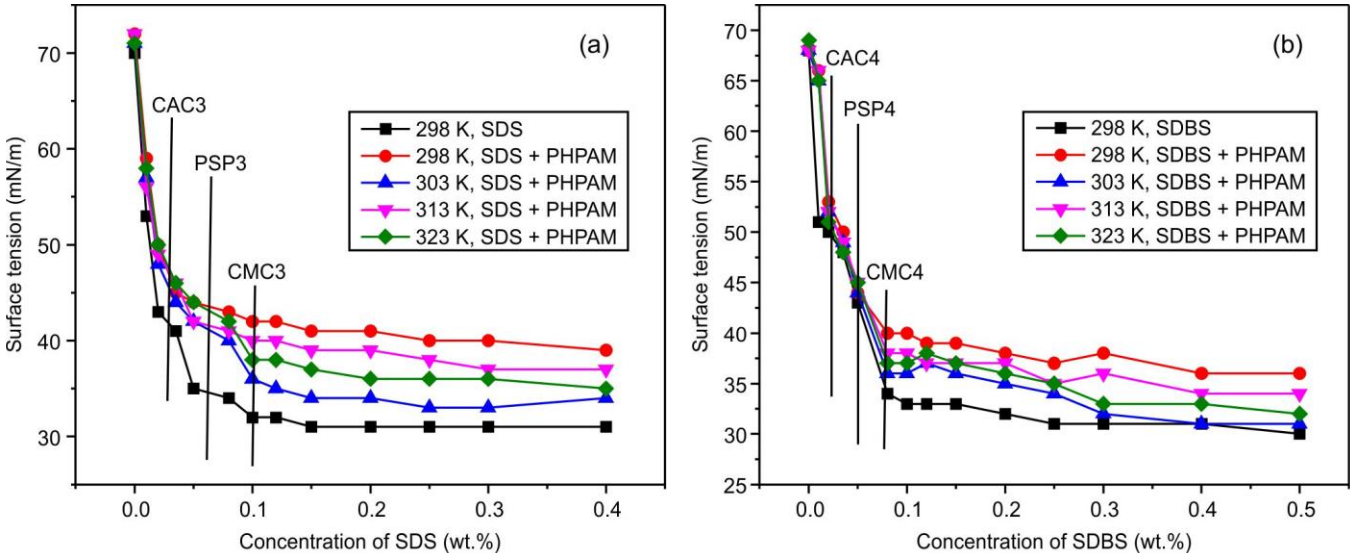

Fig. 4. Effect of PHPAM on surface tension (a) SDS and (b) SDBS at different temperatures

here, the degrees of dissociation, counterion association, universal gas constant, temperature in absolute scale and CMC in mole fraction unit are denoted by the letters α, β, R, T and X_{CMC}, respectively. Standard molar enthalpy of CMC formation was determined by putting the value of slope from ln X_{CMC} *vs.* T plot [41]. The nature of the plot is straight line with good correlation coefficient value.

Thermodynamics analysis for surfactant-polymer interaction at constant temperature (T = 298 K) and different concentration of polymers (500 ppm to 5000 ppm) has been studied and the result shows that the standard free energy values for surfactant-polymer mixture decreases (more negative) compared to the pure surfactant solution due to the formation of hydrogen bond with -OH group of non-polar guar-gum polymer molecule and negatively charged head group of SDS or SDBS, enhancing the micelle formation at lower surfactant concentration and also stabilized the complexes in mixture by hydrophobic interaction with surfactant's tails (hydrophobic) and non-polar part of polymer. Similarly, PHPAM is a synthetic and partially anionic polymer that strongly interacts with SDBS and SDS through electrostatic interaction, H-bonding and hydrophobic binding that form a more stable aggregation in mixture unto a certain concentration of polymer. At the lower concentration of polymer, available sites are there for the polymer and surfactant interaction to moderate concentration of polymer. The moderate concentration of guar-gum and PHPAM are1000 ppm and 1500 ppm respectively with SDS and SDBS at which free energy is more negative and micellization reaction is spontaneous (Fig. 5). At high concentration of polymer, entangle or overlapping (self-association) became started due to fewer binding sites and steric hindrance and hence surfactant can no longer interact with free polymer molecules, lower the free energy (less negative). The SDBS and guar-gum mixture show more negative free energy value compared to the SDS and guar-gum mixture, because bulky aromatic ring containing hydrophobic tail of SDBS formed a better packing with polymer and stabilized the aggregated complex, but the linear tail group of SDS restricted to close packing. At constant temperature the enthalpy change (ΔH^o_m) is zero during the surfactant and polymer interaction although the free energy (ΔG^o_m) of these reaction increases due to entropy driven reaction (ΔS^o_m = positive) even at constant temperature.

Similarly, standard Gibbs free energy (ΔG^o_m), standard enthalpy (ΔH^o_m) and standard entropy (ΔS^o_m) of micellization per mole of monomer, respectively were analyzed at different temperatures (298 K, 303 K, 313 K and 323 K) and at a fixed polymer concentration, 1000 ppm for guar-gum and 1500 ppm for PHPAM (Fig. 5b). In this study, all the calculated standard Gibbs free energy values are negative, which indicate that the process of micellization occurs naturally and thermodynamically favourable. The Gibbs free energy values decrease (more negative) on increasing the temperature onto a certain point. Kinetic energy of molecules increases with rise in temperature, surfactants molecules overcome repulsive forces leading to aggregation but potentially destabilizes the micelle at very high temperature due to breaking hydrophobic interaction and H-bonding.

All the standard entropy (ΔS^o_m) values are also positive *e.g.* the reaction process is spontaneous (Fig. 6), which implied that the polymer-surfactant interaction is more favourable. This study reveals that the micellization process of SDS and SDBS in presence and absence of guar-gum and PHPAM polymer is feasible and spontaneous. Temperature also plays a vital role for the aggregation formation of surfactants and polymer to a certain point then destabilization occurs, at 303 K temperature the polymer-surfactant aggregated complex is more stable due to maximum interaction and more negative free energy (Fig. 6b). The micellization process is more favourable for SDBS and PHPAM mixture (maximum free energy) than SDS and PHPAM mixture at fixed concentration of polymer due to the strong electrostatic interaction between carboxylate (-COOH) group of PHPAM and cationic part of micelles at moderately high temperature but non-ionic guar-gum only shows the hydrophobic interaction that is weaker than it. The results show that the micellization process is exothermic, having negative ΔH^o_m values (Fig. 7).

Conductometric studies: The variation of electrical conductivity with increasing surfactant concentration is depicted

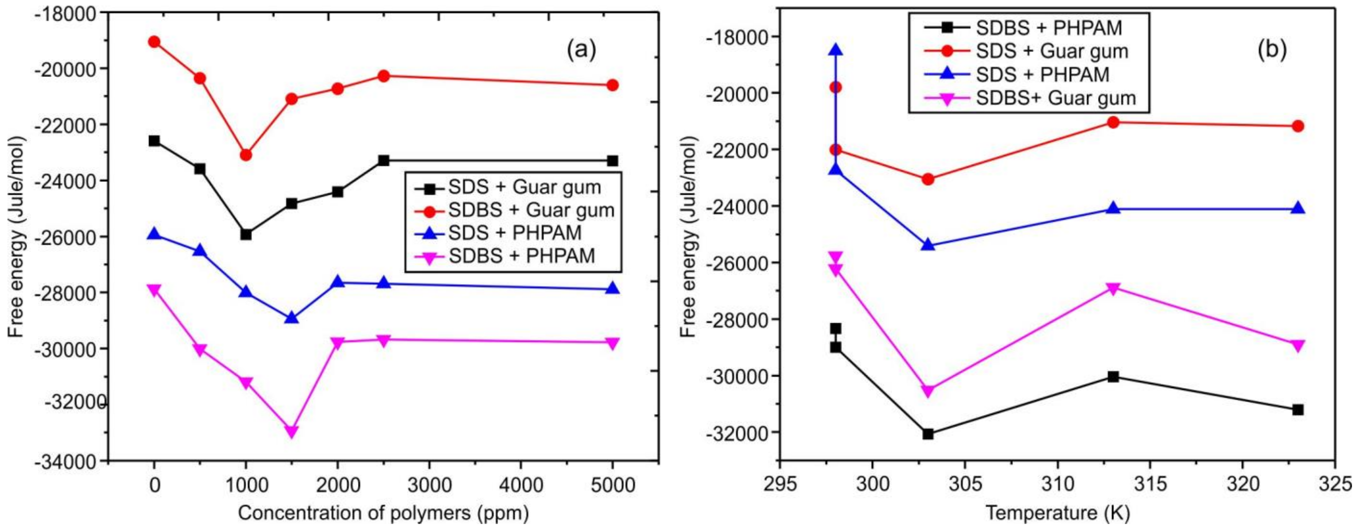

Fig. 5. Variation of standard Gibbs free energy (ΔG^o_m) of surfactant-polymer mixture at (a) 298 K and (b) different temperatures

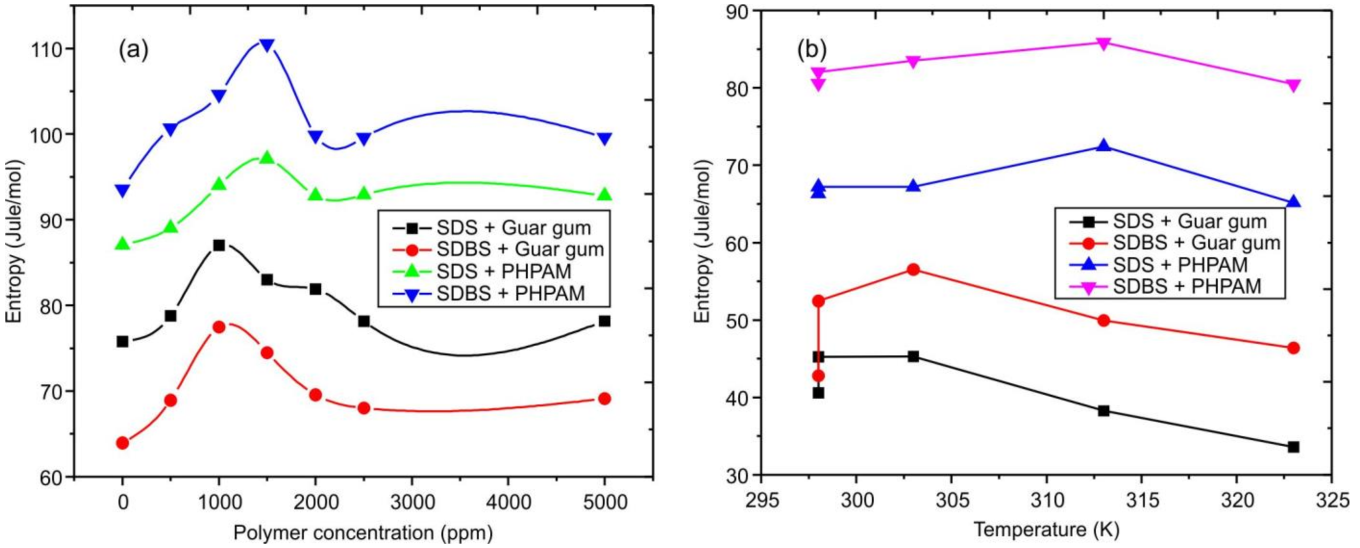

Fig. 6. Variation of standard entropy (ΔS^o_m) of surfactants-polymers mixture at (a) 298 K and (b) different temperatures

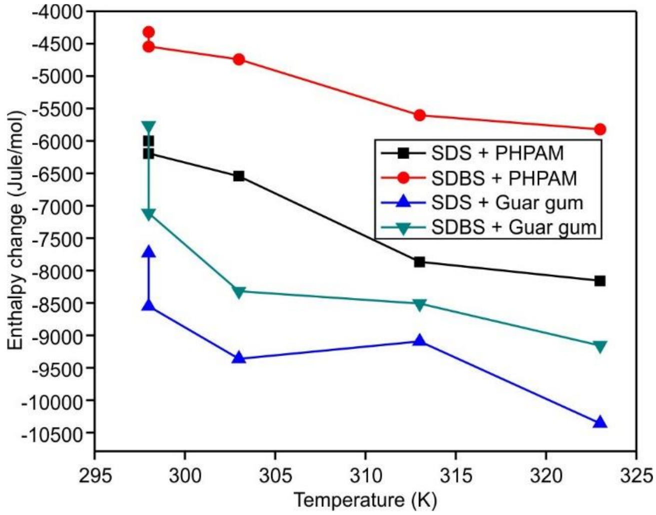

Fig. 7. Variation of standard enthalpy (ΔH^o_m) of surfactant-polymer mixture at different temperatures

in Figs. 8 & 9, both in the absence and presence of polymers. In the polymer-free systems, the conductivity of the solution increases proportionally with surfactant concentration, as the dissociation of the ionic surfactants–sodium dodecyl sulfate (SDS) and sodium dodecylbenzene sulfonate (SDBS)–produces more charge carriers, thereby enhancing the current-carrying capacity of the solution.

For SDS, the specific conductivity increases from contributions of free sodium ions (Na^+) and dodecyl sulfate anions (DS^-). Similarly, for SDBS, the conductivity results from the free sodium ions (Na^+) and dodecylbenzene sulfonate anions (DBS^-). In the presence of polymers such as guar gum or partially hydrolyzed polyacrylamide (PHPAM), the CMC shifts to the higher surfactant concentrations. This is attributed to the interaction between polymer chains and surfactant molecules, which delays micellization and alters the ion mobility. Without polymer, smaller free ions move more freely in solutions, resulting in higher specific conductivity values up to the CMC.

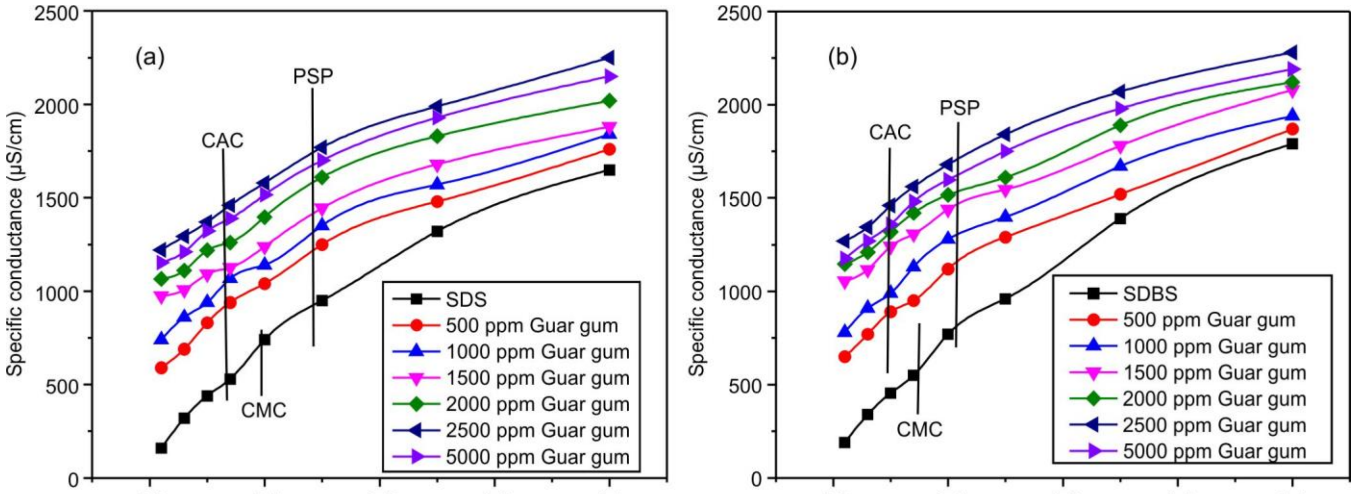

Fig. 8. Effect of guar gum on conductivity of (a) SDS and (b) SDBS at 298 K

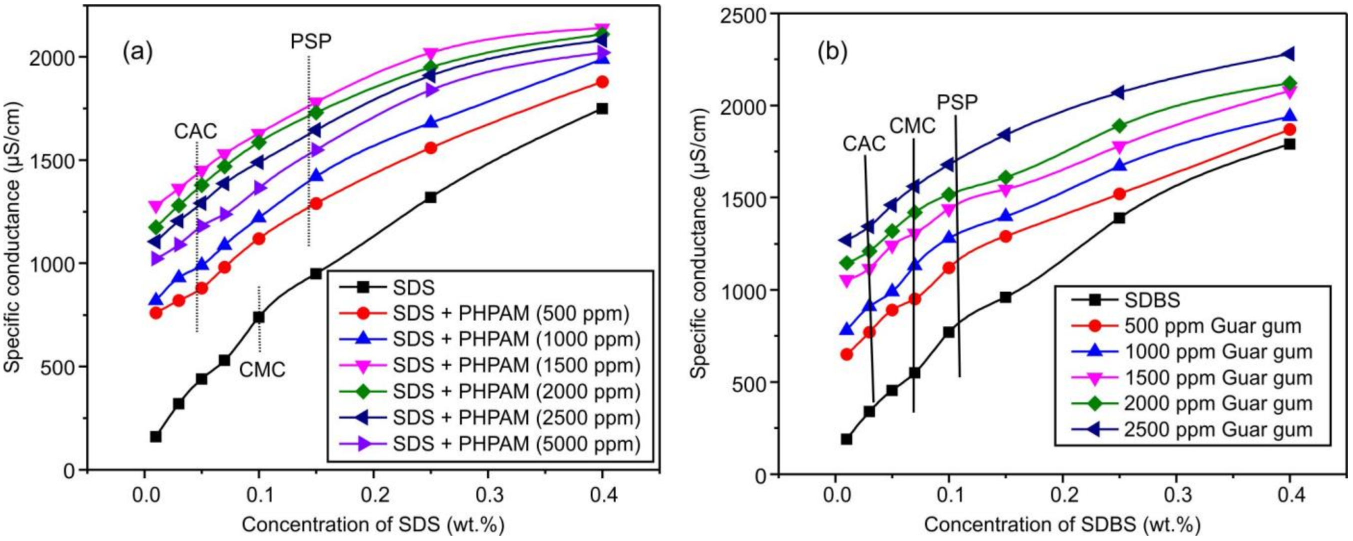

Fig. 9. Effect of PHPAM on conductivity of (a) SDS and (b) SDBS at 298 K

The total specific conductivity (κ) for SDS and SDBS systems can thus be expressed as follows:

$$\kappa_1 = \kappa_{SDS} = \kappa_{Na^+} + \kappa_{DS^-} \quad (4)$$

$$\kappa_2 = \kappa_{SDBS} = \kappa_{Na^+} + \kappa_{DBS^-} \quad (5)$$

In systems containing pure SDS or SDBS, it is commonly assumed that the surfactant molecules undergo complete dissociation into their constituent ions below the CMC. Above the CMC, changes of curve behaviour of SDBS are like SDS, the same explanation was postulated.

In the presence of guar gum, specific conductivity, κ'_1 (with SDS) and κ'_2 (with SDBS) can be calculated by eqns. 6 and 7.

$$\kappa'_1 = \kappa_g = \kappa'_{Na^+} + \kappa'_{DS^-} \quad (6)$$

$$\kappa'_2 = \kappa'_g = \kappa'_{Na^+} + \kappa'_{DBS^-} \quad (7)$$

where κ_g is the guar gum specific conductivity of the charged polymer in SDS and κ'_g is the guar gum in SDBS solutions, respectively. In contrast to the surfactant-containing curves, it is assumed that all species present in the solution below CMC had completely dissociated as the conductivity migrated to the higher areas. The total specific conductivity of the mixture can be computed using the following formula when PHPAM is present and the concentrations of polymers and surfactants are higher than the CMC (eqns. 8 and 9):

$$\kappa''_1 = \kappa_{PHPAM} = \kappa''_{Na^+} + \kappa''_{DS^-} \quad (8)$$

$$\kappa''_2 = \kappa''_{PHPAM} = \kappa''_{Na^+} + \kappa''_{DBS^-} \quad (9)$$

where κ''_1 and κ''_2 represent the specific conductivity of SDS and SDBS with PHPAM solutions respectively, while κ_{PHPAM} and κ''_{PHPAM} represent the specific conductivity of the charged polymer PHPAM in SDS and SDBS, respectively.

In presence of polymers (guar gum and PHPAM), some surfactant molecules may bind with polymers and reduce the free ions in solutions and hence the rate of increase of specific conductance is low. SDS gives higher specific conductance than SDBS in presence of guar gum and PHPAM as SDS binds less tightly and discharges more free ions into the solution. SDBS binds with polymers even more efficiently, which releases a smaller number of free ions in solution (Figs. 8 and 9). The specific conductance increases with rise in temperature as the viscosity of solution decreases and the thermal energy (ionic mobility) of solution increases thus the specific conductance for ionic surfactant (SDS and SDBS) solution linearly increases up to CMC point (Figs. 10 and 11).

Thermodynamics of micellization for conductometric investigation: It has been observed that the ΔG^o_m values are closer to the surface tension measurement process. The results suggested that stability of mixed micelles increases with increasing the high negative value of standard free energy due to the electrostatic attraction of surfactant's head group and polymers (Fig. 12). At constant temperature (T = 298 K), the SDBS–PHPAM mixture shows the more negative free energy value than other interactions as SDBS forms a strong bond with PHPAM to give a stable surfactant-polymer complex *via* hydrophobic interaction. The ΔH^o_m values are zero at constant temperature that indicates the surfactant-polymer interaction is entropy orientated although the free energy is negative. The ΔS^o_m values are comparably higher and positive, *e.g.* the processes are spontaneous (Fig. 13).

The results from the conductance measurements validate the micellization behaviour revealed by thermodynamics calculation and surface tension analysis. Conductance value first increases due to increase of concentration of surfactants leading to generating a greater number of ions in solutions. In the presence of polymer in the surfactant solution a similar shift occurs in conductance curve, CMC observed at lower concentration of surfactant. This pattern suggests greater aggregation (micellization) because of better polymer interaction with more negative free energy and positive entropy change. The conductance measurement and its thermodynamics parameters calculation, the variation of free energy, enthalpy and entropy change with the concentration of polymers are similar trends to that of surface tension measurement at a fixed temperature and different temperatures.

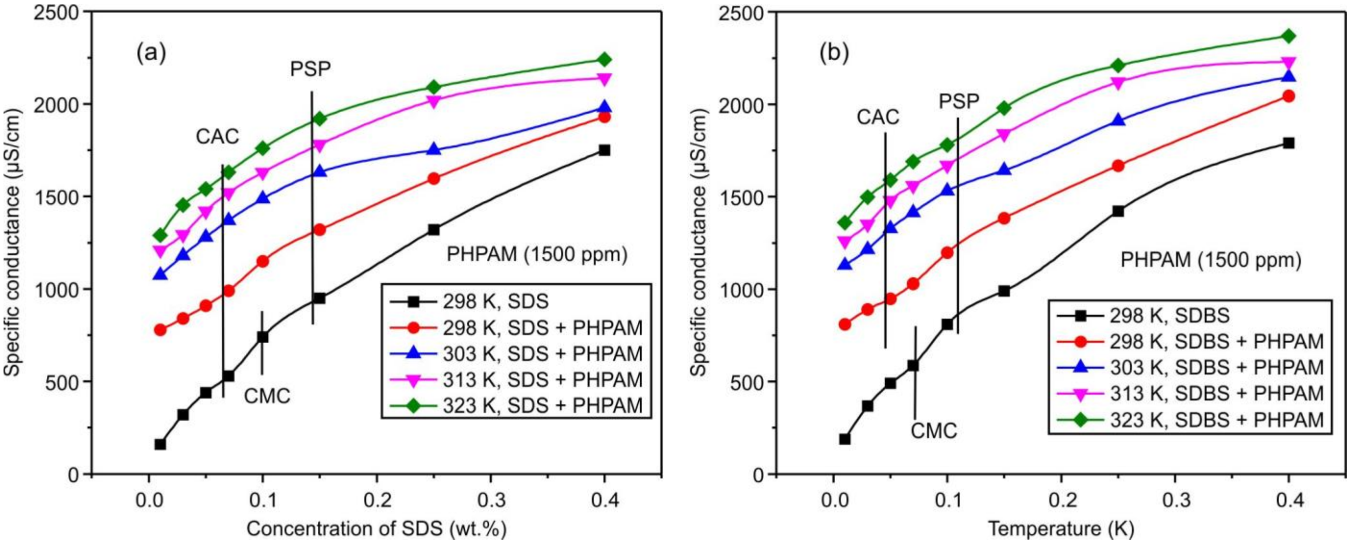

Fig. 10. Impact of PHPAM on specific conductance of (a) SDS and (b) SDBS solution at different temperatures

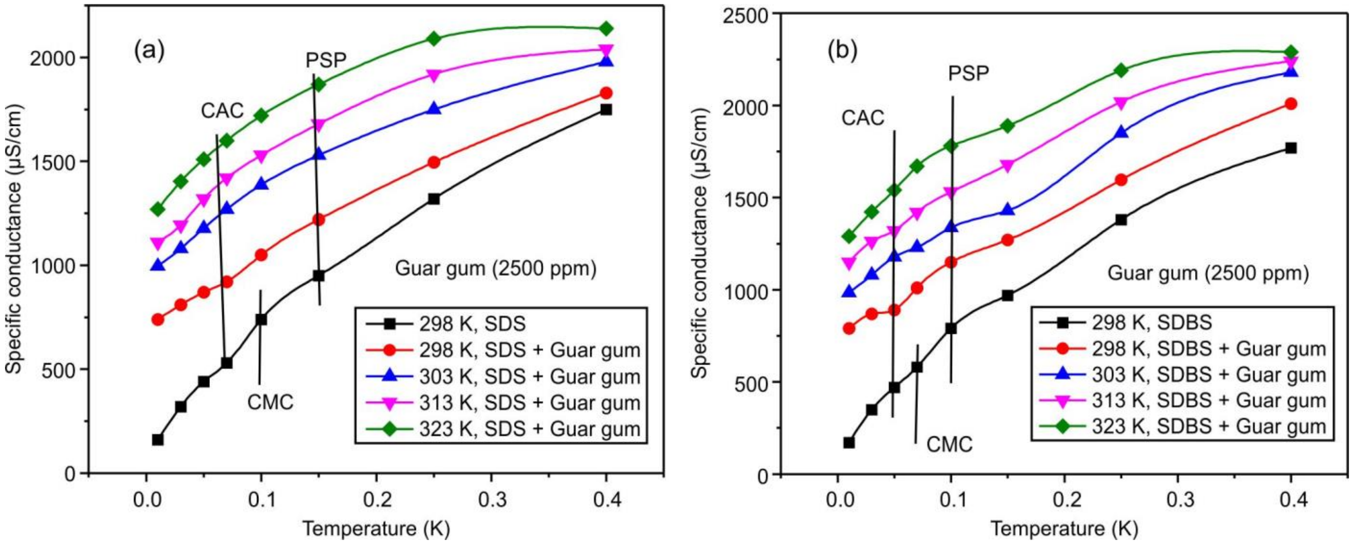

Fig. 11. Impact of guar gum on specific conductance of (a) SDS and (b) SDBS solution at different temperatures

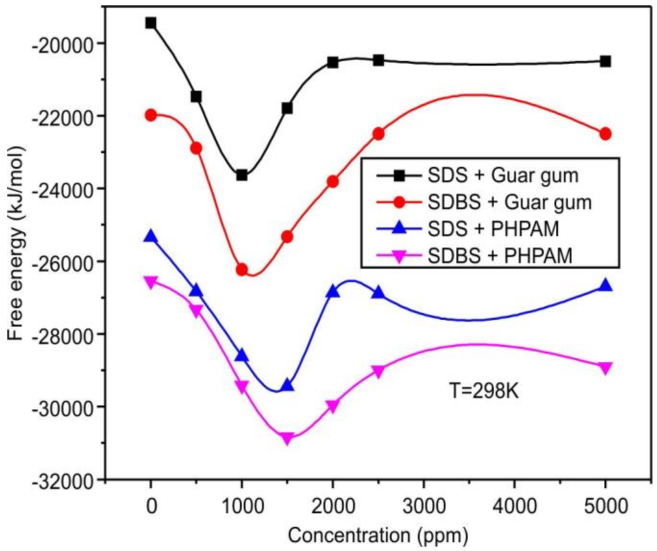

Fig. 12. Variation of standard Gibbs free energy (ΔG^o_m) for specific conductance of surfactant-polymer mixture at 298 K

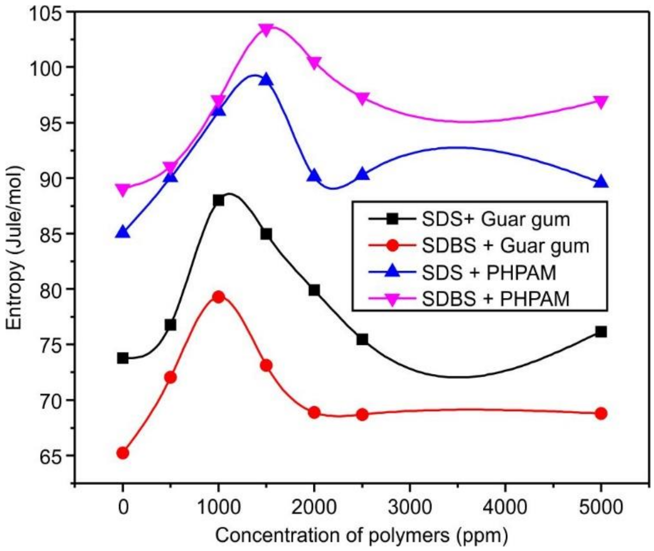

Fig. 13. Variation of standard entropy (ΔS^o_m) for specific conductance of surfactant-polymer mixture at 298 K

Conclusion

In this study, several experiments were performed at ambient and elevated temperatures to investigate the interactions of various surfactants (SDS and SDBS) and polymers (PHPAM and guar gum) in aqueous solution with different concentrations and temperatures. The variation of CMC in mixed micelles depends on the composition of mixture components. The comparatively higher stability of mixed micelles is indicated by the excess negative value of the free energy of mixing. The electrostatic interaction between ionic head groups most likely determines the stability of mixed micelles. All the calculated standard Gibbs free energy values (ΔG^{o}_{m}) are negative, indicating that the micellization process is thermodynamically spontaneous under the studied conditions. The highest magnitude of ΔG^{o}_{m} was observed for the SDS–guar gum mixture at 1000 ppm and the SDBS–PHPAM mixture at 1500 ppm, signifying greater micellar stability in these systems. The standard enthalpy of micellization (ΔH^{o}_{m}) was approximately zero at 298 K, suggested that the micellization process is entropy-driven. Consistently, all standard entropy values (ΔS^{o}_{m}) were positive, confirming the increased disorder associated with micelle formation and supporting the spontaneity of the process. The thermodynamic parameters obtained from both surface tension and conductivity analyses show a consistent and coherent trend across both methods, validating the reliability of the experimental approach. This study contributes to a deeper understanding of surfactant–polymer interactions and micellization behaviour, offering the valuable insights for applications in the formulation science, enhanced oil recovery, wastewater treatment and other industrial and environmental processes where surfactant-polymer systems play a critical role.

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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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