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ORIGINAL RESEARCH

Effect of Aprotic Solvent on the Energetics and Micellization of Quaternary Ammonium Surfactant, Benzethonium Chloride

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Abstract:

Introduction: The influence of polar organic solvent on the self-assembly of surfactants is of significant concern. Specifically, certain biological and technical processes required non-aqueous media to achieve optimal outcome.

Aims: This work investigated the effect of an aprotic solvent (acetonitrile) on the micellization of benzethonium chloride (BZC) at 298.1-318.1.K at an interval of 5 K.

Materials and Methods: The micellization of benzethonium chloride in ACN was studied with the aid of temperature controlled-water bath and electrical conductivity meter. The stock solution of BZC was prepared in redistilled-de-ionized water, and then in mixed varying volume percentages of ACN. **Results:** The values of the critical micelle concentration (CMC_{BZC}) obtained at different temperatures in aqueous medium ranged from 2.885 to 3.486 moldm^{-3}. When 5% v/v of ACN was added to the system, the values of the CMC_{BZC} were 3.093 - 3.545 moldm^{-3} at 298.1 - 318.1 K. However, when the concentration of ACN was further increased to 10% v/v and 15% v/v, the process of micellization was depressed at different temperatures. The values of the Gibbs free energy (ΔG_{BZC}, kJmol^{-1}) were all negative, while the values obtained for the ΔS_{BZC}, $Jmol^{-1}K^{-1}$, were positive with variation in the degree of randomness at different volume percentages of ACN and temperatures. In addition, the ΔH_{BZC}, $kJmol^{-1}$ values revealed that the system was on the overall, exothermic.

Conclusion: The ACN adjusted the aggregation process in the context of co-solvent, and the thermodynamic data indicated that the ACN-BZC micelle can be utilized in water-non-aqueous based system.

Keywords: Benzethonium Chloride, Thermodynamic Parameters, Acetonitrile, Mixed solvent

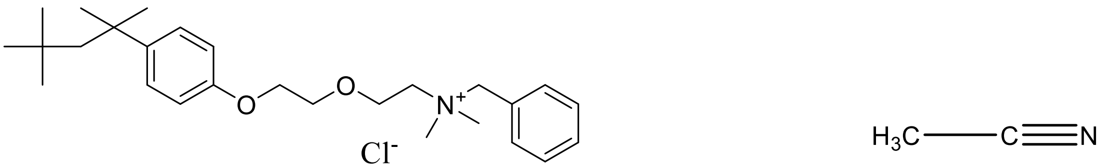
1. INTRODUCTION

Surfactants play a vital role in modern life, owing to its versatility, and widespread applications across various processes [1]. They are amphiphilic in nature, and the lipophilic portion is sequestered away from the aqueous medium, while the hydrophilic portion points inside water. This characteristic is responsible for their capacity to decrease the surface tension of water [2-5]. Surfactants exhibit a wide range of structural classification, which primarily is dependent upon whether the head group possesses a net charge (ionic)) or not (non-ionic). Nonetheless, the tendency to aggregate into an organized molecule (micelle) at a well-defined concentration, known as the critical micelle concentration (CMC) remains a fundamental property [6 -8]. The efficacy of surface-active-based products is largely contingent upon the values of the CMC as the micelle formation must occur prior to using the medium for solubilization purposes. The surface activity and aggregation process are susceptible to modification by a range of environmental factors such as non- aqueous solvents, co-solute, temperature, and electrolytes [9]. The consequences of the stimuli concerning the aggregation event are determined by its effect on the product formation (micelle) with decrease or increase in the values of the CMC corresponding to reinforcement or inhibition of the micellar formation [9].

Benzethonium Chloride (BZC) (Figure 1a) is a white odorless salt that belongs to the group of quaternary ammonium surfactant [10]. It possesses aromatic rings in its structure and has numerous applications of biological importance, notably as anti-viral, antiseptic and anti-fungi agents, showcasing its potential in various therapeutic areas [11]. In addition to its potent biocidal activities at the pre-micellar concentration activities, benzethonium chloride can also be used as an inhibitor of N-80 carbon steel [12-14].

Acetonitrile (ACN) is a polar organic solvent used for organic synthesis and purification. Application of non-aqueous solvents such as acetonitrile is an important process for surfactant adsorption and micelle formation. This is because the variation in solvent types can alter the dynamic adsorption of surfactant monomers at surfaces / interfaces. This occurs as a result of solvophobic or solvophilic interaction and has a marked effect on the physicochemical properties of the resulting micelle [9, 13, 15, 16]. Organic-based surfactant systems play a crucial role in numerous processes that require specialized media such as non-aqueous solvents, particularly in pharmaceutical formulation [17], lubrication, detergency, catalytic micellar reactions and applications [18, 19], micellar enzymology [20], and oil film cleaning. A lot of work has been reported in the literature on the effect of co-solvent, co-solute and electrolytes on the micellization process and aggregation pattern of ionic and non-ionic surface active agents [18, 21]. Studies of the effect of different types of non-aqueous solvents such as acetonitrile (figure 1b) which are driven by practical and technological application are scanty [19, 22] (Figure 1b]. Specifically, it is of immense significance to the optimization of several vital processes such as drug delivery, emulsion stability, and enzyme catalysis [18-19].

Hence, understanding the micellization process of benzethonium chloride, and its thermal stability particularly in mixed aqueous-organic solvent systems, is of significant interest due to its potential implications across various fields [10-14]. This work, therefore, investigated the effect of acetonitrile (ACN) on the micellization of benzothonium chloride (BZC) at 298.1 – 318.1 K at an interval of 5 K.



(a) (b)

Figure 1: Structures of (a) benzethonium Chloride (b) acetonitrile

2. MATERIAL AND METHODS

The experimental reagents, benzethonium chloride (BZC) and acetonitrile (≥ 99 purity) were procured from Sigma-Aldrich and used without further purification. Redistilled-de-ionized water, with a specific conductance value of 1-3 μScm^{-1} at 298.1 K was used for the preparation of the solutions. A standard solution of potassium chloride (0.01 N) of conductivity value of 1413 μScm^{-1} was used to calibrate the conductivity meter (Jenway 4510) prior to the commencement of the experiment. The stock solution of BZC was prepared in redistilled-de-ionized water, and then in mixed varying volume percentages of ACN. A 10 cm^{-3} of water or the appropriate solvent was initially placed in the cuvette, this was followed by the progressive addition of 0.2 cm^{-3} aliquots of the stock solution (2.8x10^{-3} moldm^{-3}) until the final volume was achieved. This was observed when the difference between the values of the successive electrical conductivity results became minimal, indicating the depletion of ions in the solution due to the formation of charged micelles.

The conductance values were recorded after every addition of the stock solution and after thorough mixing. This procedure was repeated with freshly prepared solutions (working solutions), and the experiment was performed at five different temperatures (298.1, 303.1, 308.1, 313.1, and 318.1 K) with the aid of temperature controlled-water bath. The conductance data were plotted against the concentration of the benzethonium chloride (BZC), and the values of the critical micelle concentration was obtained at the inflection point of the graph as shown in Figure 2.

3. RESULTS AND DISCUSSION

3.1 Critical micelle Concentration (CMC_{BZC}) and the fraction of counter-ion (Cl^-) dissociation (α)

The values of the critical micelle concentration (CMC$_{BZC}$) and the counter-ion dissociation (α) calculated for the system is presented in Table 1. Similarly, the graph of conductance against the concentration of

BZC in aqueous medium at different temperatures is depicted as Figure 2 and the break point in the plot corresponded to the CMC_{BZC}. The ratio of the slopes of the post-micelle(S_2) and pre-micelle (S_1) section in the graph (Figure 2) corresponds to the degree of counter-ion dissociation(α); $\left(\alpha = \frac{S_2}{S_1}\right)$.

Table 1: Critical micelle concentration (CMC_{BZC}) and Counter-ion Dissociation (α) for Benzethonium Chloride in aqueous and various Percentages of Acetonitrile (ACN) at different Temperatures.

System	Solvent v/v(%)	$\frac{Temperature}{K}$	$\frac{CMC_{BZC}\,X10^{-3}}{moldm^{-3}}$	α
Water	0.0	298.1	2.885	0.424
		303.1	2.887	0.466
		308.1	3.038	0.440
		313.1	3.207	0.436
		318.1	3.486	0.407
Water+ Acetonitrile	5	298.1	3.093	0.496
		303.1	3.195	0.502
		308.1	3.321	0.488
		313.1	3.268	0.428
		318.1	3.545	0.442
Water+ Acetonitrile	10	298.1	3.531	0.563
		303.1	3.533	0.483
		308.1	3.376	0.488
		313.1	3.469	--
		318.1	--	
Water+ Acetonitrile	15	298.1	4.095	0.540
		303.1	4.516	0.451
		308.1	3.767	0.498
		313.1	3.510	0.655
		318.1	--	--

The CMC_{BZC} values obtained at 298.1 K was $2.885\ moldm^{-3}$, and this value showed similarity to those previously reported in the literature [13]. However, a linear increase in the values of the CMC_{BZC} was obtained as the temperature increased to 303.1, 308.1. 313.1, and 318.1 K respectively. This behaviour can be associated to two opposing forces. Firstly, significant hydration of the head group of the monomeric benzothonium chloride would result into vigorous interaction of hydrophobic group (the driving force for micelle formation) and this favors micelle formation. Secondly, at elevated temperature, the 3-D structure of water got disrupted which resulted into a decline in the hydrophobic interaction, and as a consequence the process of micellization would be elongated and a concomitant increase in CMC of the system [23]. This behavior is peculiar to ionic surfactants and it is well reported in the literature [24-25]. In this work, the second factor was dominant as the CMC_{BZC} increases as the temperature was increasing. The degree of counter ion dissociation was included in Table 1 and it was found to be independent of temperature.

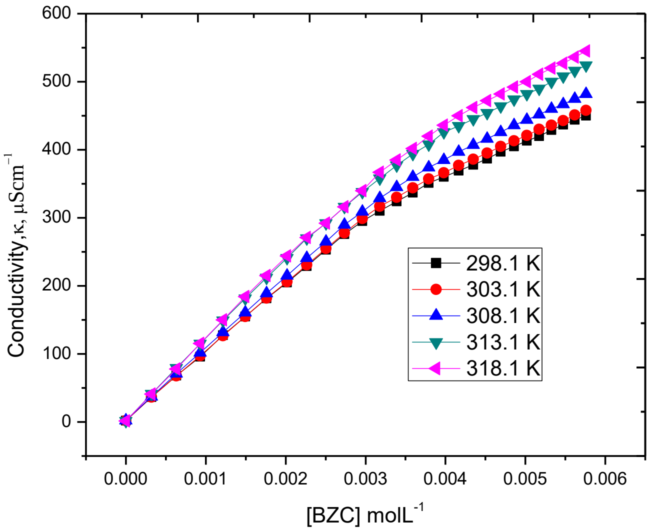


Figure 2: Specific conductance against the concentration of BZC in aqueous medium at different temperatures.

The addition of 5 v/v % acetonitrile (ACN) led to a further increase in the values of CMC_{BZC}, and the values were 3.093, 3.195, 3.221, 3.268, and 3.545 $moldm^{-3}$ at 298.1, 303.1, 308.1, 313.1, and 318.1 K respectively. A further increase in the values of the critical micelle concentration was observed when the system was accentuated with 10 v/v%, of the solvent. The values increased as the temperature was increased up to 303.1 K, it then decreased and increased again. At 15 v/v% addition, an increase in the values of the CMC_{BZC} was observed with rise in temperatures, and a trend similar to the one obtained at 10 v/v% was observed (Table 1). The changes in the value of CMC_{BZC} (χ_{CMC}) is graphically represented in Figure 3 as a function of temperature. The increase in CMC_{BZC} as the temperature was increasing at a particular concentration (v/v) of ACN suggested that the monomeric BZC was stabilized in ACN solution. ACN is a water structure breaker, therefore, there was an overall increase in the electrostatic repulsion of the head group (hydrophilic group and a delayed in the micelle formation was noticed through the values of the CMC_{BZC}.

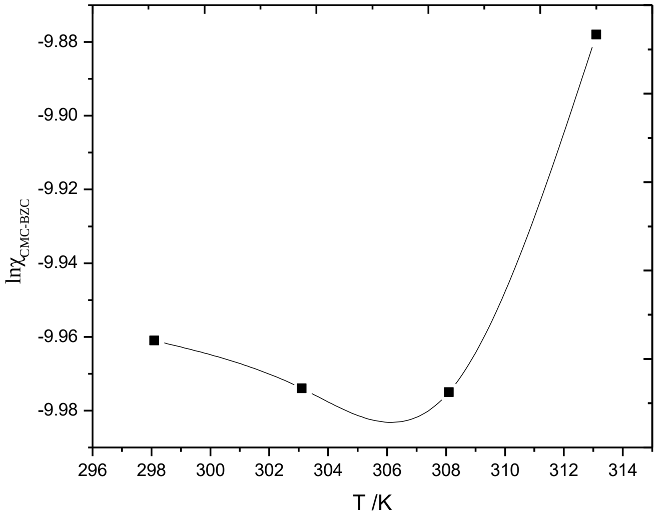


Figure 3: Representative plot of $\ln\chi CMC_{BZC}$ against temperature at 10% v/v

Also, it was observed that the value of the CMC_{BZC} increased as the percentage concentration of ACN was increasing. This is not unconnected with solvophobic forces and hydrophilic / lipophilic interactions. The dielectric constant of the medium decreased with increasing percentage of ACN [26] and this also caused an increase in the electrostatic repulsion among the head groups of BZC at higher concentration of ACN [27]. As a consequence, a delay in the micelle formation and an increase in the values of the CMC_{BZC} was discovered at higher temperatures and concentration as indicated in the inflexion point of the plots of conductivity versus concentration at different temperatures (Figure 2). The U-shaped (Figure 3) behaviour was an indication of hydrophobic and hydrophilic hydration at high temperatures [24, 28].

3.2 Thermodynamic Parameters of Micellization of Benzothonium Chloride

The critical micelle concentration, CMC_{BZC}, is susceptible to changes in temperatures (Figures 2 and 3) as a result of the extent of hydration of the surfactant monomers at elevated temperatures [26, 27]. This offers valuable information concerning the stability of the micelles in various industrial processes [18]. The thermodynamic parameters were obtained by employing Equations 1 - 4 and the summary of the results are presented in Table 2.

The change in Gibbs free energy (ΔG_{BZC}) of the benzothonium micelle was obtained from equation (1a) in agreement with the pseudo- phase separation model [9], which put into account the transfer of $1-\alpha$ moles of counter ion (Cl^-) alongside the benzethonium molecules into the micellar phase.

$$\Delta G_{BZC} = (2-\alpha)RT\ln\chi cmc_{BZC} \qquad \text{Equation (1a)}$$

Where $\chi_{CMC\ BZC}$ is the CMC of the micelle expressed in mole fraction, and α is the degree of counter-ion dissociation, R is the gas constant and T is temperature.

The transfer of the tail group of the BZC surfactants in the mixed solvent can be obtained by the following expression:

$$\Delta G_{BZC\,(trans)} = \Delta G_{BZC+water} - \Delta G_{BZC\,(water)} \qquad \text{Equation (1b)}$$

Table 2: Thermodynamic Parameters of the Micelles of Benzethonium Chloride (BZC) containing different Concentration of ACN

System	**Solvent (v/v%)**	$\frac{Temperature}{K}$	$\frac{-\Delta G_{BZC}}{kJmol^{-1}}$	$\frac{-\Delta H_{BZC}}{kJmol^{-1}}$	$\frac{\Delta S_{BZC}}{Jmol^{-1}K^{-1}}$	$\frac{\Delta G_{TRANS}}{kJmol^{-1}}$
Water	0.0	298.1	34.818	-1.662	122.379	
		303.1	36.179	3.341	108.337	0.0
		308.1	36.200	8.239	90.752	
		313.1	36.693	13.169	75.132	
		318.1	36.009	17.752	57.395	
Water+		298.1	37.425	3.913	112.420	2.606
acetonitrile	5	303.1	38.081	5.495	107.508	1.903
		308.1	38.199	6.999	101.268	1.999
		313.1	37.553	8.269	93.529	0.859
		318.1	37.972	9.802	88.558	1.963
		298.1	38.586	-6.813	152.296	-3.767
Water +		303.1	37.222	-3.625	134.762	-1.044
Acetonitrile	10	308.1	37.524	-8.088	124.416	-1.324
		313.1	38.471	2.052	116.318	-1.778
		318.1	--	-	-	--
	15	298.1	37.454	18.545	63,428	-2.634
Water +		303.1	35.523	-15.430	135.115	0.655
Acetonitrile		308.1	37.973	-29.438	218.807	-1.777
		313.1	43.064	-58.676	224.946	-6.371
		318.1		-	-	--

The parameter, $\frac{\delta \ln \chi_{CMC\ BZC}}{\delta T}$ was obtained by plotting $ln\chi\, CMC_{BZC}$ against temperature using the appropriate order of derivative (Equation 2), to obtain the slopes of the graph at different temperatures.

$$ln\chi\, CMC_{BZC} = A_0 + A_1 lnT + \frac{A_2}{T} \qquad \text{Equation (2)}$$

The entropy change associated with the process was then obtained using Equation 3:

$$\Delta S_{BZC} = (\Delta H_{BZC} - \Delta G_{BZC})T^{-1} \qquad \text{Equation (3)}$$

The values of Gibbs free energy of the micelle of BZC were negative in aqueous medium, and the values became more negative at elevated temperatures as shown in Table 2. The values of ΔG_{BZC} in 5 v/v% ACN were more negative with values that ranged from -37.425 $kJmol^{-1}$ at 298.1 K to -38.199 $kJmol^{-1}$ at 308.1 K, which is an indication of increased spontaneity. When the percentage content of the solvent was increased to 10 and 15%, the micellization process remained spontaneous (Table 2). The overall increase in the values of ΔG_{BZC} was an indication of delayed in the aggregation process as earlier observed from the results of the CMC_{BZC}. Furthermore, the results of the Gibbs free energy of transfer $\Delta G_{BZC\ (trans)}$ (Table 2) showed that the effect of ACN (5 v/v% on the micellar formation was positive at various temperatures as a result of decrease in lipophilicity-solvophobicity of the system which later became weakly negative as the concentration of ACN was increasing. Nevertheless, the transfer process from the bulk phase was energetically viable [16, 30].

The results of the enthalpy of the process were also included in Table 2. The heat of the micelle formation was negative in aqueous medium except at 298.1 K. At 5% addition of ACN, the system was exothermic at all the temperature investigated. However this trend was different at higher concentration of ACN as the system was both exothermic and endothermic due to London dispersion forces [26], and in the same manner the reinforcement of the hydrogen bonding of water.

The entropy of the system was evaluated using Equation 3, and summarized in Table 2. The values of the entropy, ΔS_{BZC}, of the micelle formation were positive in aqueous and in co-solvent with varying degree of randomness on increasing the content of ACN. This is the consequence of the liberation of water molecules during the formation of micelle and continuous breaking and formation of hydrogen bonding [24]. Nonetheless, the process was favored by entropic gain.

3.3 Determination of Enthalpy–Entropy Compensation

Further examination of the micelle of BZC was also investigated by evaluating the relationship between the heat of reaction and the degree of randomness (enthalpy- entropy compensation) of the system. The possibility of obtaining a linear relationship or otherwise [31, 19] between the change in enthalpy and entropy (Figure 4) was investigated in accordance with Equation 4:

$$\Delta H_{SYS} = T_c \Delta S_{SYS} + \Delta H^* \qquad \text{Equation (4)}$$

The values of the compensation temperature (T_c) and the intrinsic enthalpy gain (ΔH^*) were obtained from the slope and intercept of the graph respectively and the results are presented in Table 3.

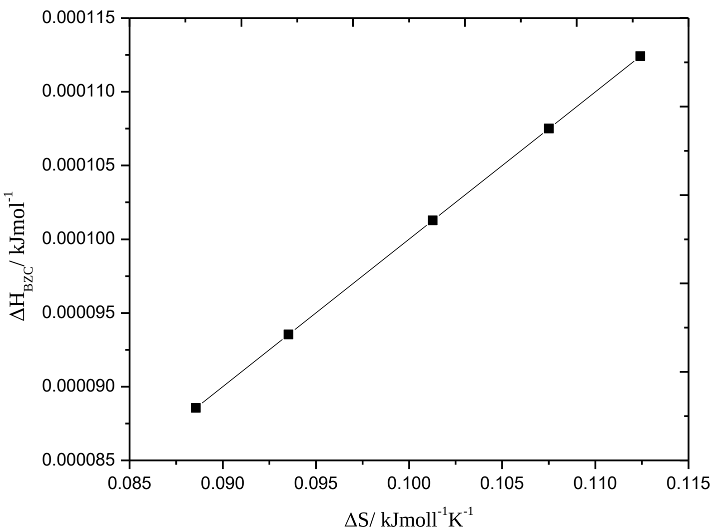


Figure 4: A typical plot of enthalpy-entropy of compensation for the micellization of BZC in aqueous co-solvent system

The enthalpy- entropy compensation plot was linear at all temperatures and co-solvent investigated with a typical plot shown in Figure 3. The slope of the graph gave the compensation temperature, T_c, with values that ranged from 233K to 297 K. T_c is an indicator of the composition phenomenon due to desolvation of the tail group of the surfactant BZC [32]. The values of ΔH^* which is associated with interactions between solute-solute and solute-solvent of micellar formation [33] were all negative with a slight variation as the concentration of ACN changes in the system. Furthermore, the values of ΔH^*correspond to the zero entropy ($\Delta S_{sys} = 0$) gain [33]. Hence, the results showed that the system was stable at the investigated temperatures.

Table 3: Enthalpy-Entropy compensation parameters for benzethonium chloride containing different concentrations of ACN

T_c	ΔH^* (kJmol^{-1}	R^2
	Benzethonium+ 0.0 Acetonitrile	
297.64	35.193	0.998
	Benzethonium + 5% v/v Acetonitrile	
233.94	30.443	0.988
	Benzethonium + 10% v/v Acetonitrile	
241.79	29.605	0.977

	Benzethonium + 15% v/v Acetonitrile	
293.39	35.696	0.998

4. CONCLUSION

The effect of acetonitrile (ACN) was investigated on the micelles of benzethonium chloride. The experimental results indicated a delay in micellization process at elevated temperatures and concentration of ACN as observed in the values of the CMC_{BZC} due to hydration effect. The formation of the micelles in water and different concentration of ACN were energetically favourable as all the values of the ΔG_{BZC} were less than zero ($\Delta G_{BZC} < 0$). Furthermore, the benzethonium Chloride-ACN micelles were formed by significant entropic gain. Overall the solvent, ACN, behaved as a co-solvent in the micellar formation of benzethonium chloride at all investigated temperature. In summary, the benzethonium-ACN micelles were thermodynamically spontaneous and feasible; consequently, the BZC micelle in ACN can be used for processes that require specialized media such as non-aqueous or solvent-based media environment (pharmaceuticals and catalysis).

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COMPETING INTERESTS

We declared that there is no conflicting interest in the publication of this study.

AUTHORS' CONTRIBUTIONS

Osundiya MO was involved in the conceptualization, methodology and writing of the original draft of the manuscript. Sobola AO was involved in the formal writing of the manuscript: review and editing, Taiwo RO was involved in methodology and formal data analysis, Oyewole TS was involved in methodology and formal data analysis, Egbemhenghe AU was involved in methodology and formal data analysis, Osifeko OL was involved in in methodology and formal data analysis. Olowu RA was involved in the conceptualization and methodology of the study.

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