



RAPID COMMUNICATION

Evaluation of micellar properties of sodium dodecylbenzene sulphonate in the presence of some salts

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Abstract. The influence of some salts (NaCl, KCl, NH₄Cl and MgCl₂) on the critical micelle concentration (CMC) and fraction of counterion dissociation (α) of sodium dodecylbenzene sulphonate (SDBS) have been determined by conductometric and dynamic light scattering (DLS) methods at 298.15 K. The CMC determination involves fitting of experimental conductivity-surfactant concentration data into the integral form of Boltzmann-type sigmoidal equation. The procedure provides much better results compared to the conventional and differential methods for surfactant such as SDBS that exhibits a gradual transition from pre-micellar to the post-micellar region. The decrease in CMC of SDBS was found to be the highest in the presence of MgCl₂ and least in the case of NaCl among the studied salts. The fraction of counter ion dissociation decreases sharply at lower concentrations of the salts (except NaCl) which have been discussed in terms of effective ionic charge on the micelles. Using CMC and α value, the aggregation number of the micelle, micellar surface area and packing parameter has been calculated and were seen to agree well with the corresponding literature values obtained by using fluorescence and surface tension measurements. The effect of salts on the change in micellar size and the surface charge has also been evaluated with the help of DLS experiments and transmission electron microscopy (TEM) images.

Keywords. Sodium dodecylbenzene sulphonate; critical micelle concentration; salt effect; conductivity; dynamic light scattering.

1. Introduction

It is well-established that critical micelle concentration (CMC) is the most significant parameter and is usually determined by the point of intersection of the two straight lines in conductivity-concentration plots.¹⁻⁴ In such situations, the conductance is taken as directly proportionate to the concentration of the surfactant by ignoring the inter-ionic interactions. However, such procedures present difficulties for plots exhibiting weak curvatures, for instance, ionic surfactants in non-aqueous solvent systems.^{5,6} Therefore, in the present work, a more accurate method, proposed by Carpena *et al.*,⁷ has been used to analyze conductivity-concentration data. It is based on fitting conductivity as a function of surfactant concentration to the integral form of Boltzmann equation as described in the Experimental section. The best fit provides the values of CMC and fraction of counter ion dissociation. This approach

offers accurate results with less errors than the conventional, differential conductivity method for systems that exhibit a gradual transition from the pre-micellar to the post-micellar region.

The investigations on the micelle formation in the presence of salts are of special interest to the scientific world⁸⁻¹⁴ as these salts form the basis of many chemical processes. The salts normally assist micelle formation and influence the energetics of the process. Further, in case of ionic surfactants, the effect of added salts on micellar behaviour is dependent on the counter ions.^{15,16}

Although many studies have been devoted to the effect of commonly used salts on such behaviour, such reports on sodium dodecylbenzene sulphonate (SDBS) are rare.¹⁷⁻²⁰ Moreover, the relative effect of different salts on micellar properties of SDBS is missing in these studies. In addition, many researchers have reported the fraction of counter ion dissociation (α) of the ionic micelles but there is no unified theory on the variation in α with salt concentration. These conflicting trends encouraged us to investigate the factors responsible for

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the behavior of SDBS solutions in the presence of different types of salts. The main objective of the study is a precise measurement of CMC and α value with the help of iterative method. These values have further been utilized to calculate aggregation number and surface area per head group of the micelles. Results thus obtained were found to be in good agreement with the literature reports.²³

2. Experimental

2.1 Chemicals

The anionic surfactant SDBS (99%) was obtained from Sigma Aldrich (USA) and used as such without further purification. All the salts NaCl (99.9%), NH₄Cl (99.0%), MgCl₂ (98.0%) and KCl (99.0%) were received from SD-Fine, Mumbai and recrystallized twice before use.

2.2 Instruments and Methods

2.2a Conductivity: The conductivity was measured using microprocessor-based conductivity meter (Systronics-306, India). The solutions were kept at constant temperature (298.15 K) by circulating water through the glass jacket surrounding the conductivity cell. Double distilled or deionized water was used for the preparation of all the solutions. The CMC was determined by fitting the specific conductivity, κ , and [surfactant], x , to the following equation.¹⁷

$$\kappa(x) = \kappa(0) + A_1x + \Delta x (A_2 - A_1) \ln \frac{1 + e^{(x-x_0)/\Delta x}}{1 + e^{-x_0/\Delta x}} \quad (1)$$

Here, the specific conductance of the solution is $\kappa(0)$ when $x = 0$; the pre- and post-micellar slopes are represented by A_1 and A_2 , respectively, and the width of transition about central point, x_0 , representing CMC is given as Δx . The data has been fitted by initially guessing the A_1 , A_2 , x_0 , and Δx in Eq. 1 to obtain an approximate value of specific conductivity, $\text{approx} \kappa_x$, corresponding to each surfactant concentration. Chi-square, χ^2 , has been defined as,

$$\chi^2 = \sum_{i=1}^n (\kappa_i - \text{approx} \kappa_x)^2 \quad (2)$$

Here, n is the number of data points, the experimental and approximate conductivity at a given total surfactant concentration are denoted by κ_i and $\text{approx} \kappa_i$, respectively. The best estimate of the model parameters (A_1 , A_2 , x_0 , and Δx) is given by the minimum value of χ^2 . Ionic micelles bind sufficient amount of counter ions in order to stabilize the self-aggregated surfactant system or the micelles by neutralizing the charge on the micelle. It lowers the intermicellar repulsion potential which results in a sharp decrease in κ at the onset of micellization leading to the break in the κ versus [surfactant] plot. The degree of counter ion dissociation (α) for ionic micelles is evaluated from the ratio of the postmicellar and premicellar slopes ($\alpha = A_2/A_1$), following the procedure proposed by Evans.²¹

2.2b Dynamic Light Scattering (DLS): The hydrodynamic diameter (D_h) and zeta potential (ζ) were obtained from DLS measurements performed at 298.15 K on Zeta-Sizer Nano-ZS light scattering apparatus (Malvern Instruments, UK) equipped with He-Ne laser in backscattering mode at a scattering angle of 173°. The samples were filtered using a membrane filter with a pore size of 450 nm in order to eliminate contamination.

2.2c Transmission Electron Microscopy (TEM): TEM imaging was carried out using a JEM-2100 electron microscope at a working voltage of 200 kV. A drop of a freshly prepared micellar solution of SDBS was placed on a carbon-coated copper grid (300 mesh) and the residual solution was blotted out. The sample was then dried in air for 24 h. Both DLS and TEM measurements were made on samples prepared with micellar solution at a concentration twice the observed CMC of the mixed system.

3. Results and Discussion

3.1 Critical Micelle Concentration (CMC)

The CMC value of SDBS was obtained from the κ versus [SDBS] plots (Figure 1) as explained above and were seen to agree well with the literature.^{22,23} In order to obtain the effect of various salts on micellar properties, the CMC values in presence of these salts have also been determined and are presented in Table 1. The error in CMC measurement thus occurred was calculated to be $\pm 5 \times 10^{-2}$ mmol dm⁻³. For each salt, the dependence of CMC on [salt] was fitted using the following relation:²⁴

$$y = \frac{(a + bx)}{(1 + cx)} \quad (3)$$

Here, y is CMC and x is [salt] in mol dm⁻³. In the case of NaCl, the experimental data has been fitted to the equation where,

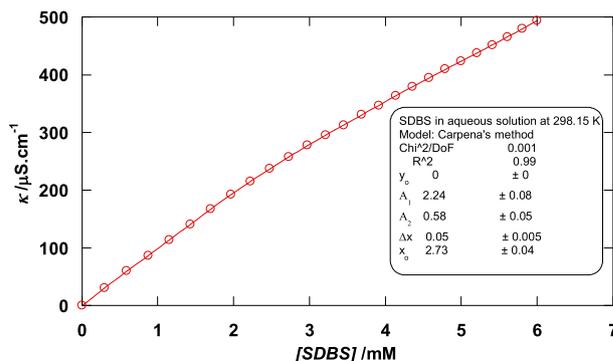


Figure 1. Specific conductance versus concentration plot (Fitted line) for SDBS at 298.15 K.

Table 1. Micellization parameters of SDBS in presence of salts at 298.15 K.

Salt	[Salt]/mM	CMC/mM	α	$\lambda_o^-/S\text{ cm}^{-2}\text{ mol}^{-1}$	
NaCl	0.00	2.73 ± 0.04 (2.58) ^a (2.90) ^b	0.52 ± 0.06	138.45 ± 0.79	
	0.50	2.14 ± 0.04	0.55 ± 0.04	143.68 ± 0.82	
	1.00	1.78 ± 0.02	0.58 ± 0.07	152.14 ± 0.67	
	2.00	1.62 ± 0.04	0.60 ± 0.03	154.37 ± 0.32	
	3.00	1.53 ± 0.03	0.63 ± 0.04	163.49 ± 0.88	
	4.00	1.47 ± 0.05	0.61 ± 0.06	158.47 ± 0.96	
	5.00	1.42 ± 0.01	0.56 ± 0.05	150.32 ± 1.02	
	7.50	1.37 ± 0.02	0.49 ± 0.04	147.61 ± 1.04	
	10.00	1.34 ± 0.03	0.38 ± 0.04	144.94 ± 0.76	
	NH ₄ Cl	0.50	1.91 ± 0.04	0.44 ± 0.02	129.64 ± 0.58
1.00		1.60 ± 0.03	0.37 ± 0.04	120.33 ± 0.62	
2.00		1.47 ± 0.02	0.39 ± 0.03	117.46 ± 0.47	
3.00		1.40 ± 0.05	0.47 ± 0.05	131.25 ± 0.88	
4.00		1.31 ± 0.01	0.53 ± 0.03	146.62 ± 0.64	
5.00		1.25 ± 0.03	0.55 ± 0.04	151.44 ± 0.96	
7.50		1.20 ± 0.04	0.46 ± 0.04	138.57 ± 0.97	
10.00		1.16 ± 0.02	0.41 ± 0.06	129.61 ± 1.02	
MgCl ₂		0.50	1.54 ± 0.03	0.59 ± 0.05	162.14 ± 0.76
		1.00	1.31 ± 0.02	0.41 ± 0.07	155.47 ± 0.58
	2.00	1.22 ± 0.01	0.36 ± 0.03	146.38 ± 0.37	
	3.00	1.10 ± 0.01	0.33 ± 0.04	139.74 ± 0.48	
	4.00	1.01 ± 0.02	0.29 ± 0.04	133.21 ± 0.95	
	5.00	0.96 ± 0.02	0.30 ± 0.06	122.25 ± 0.84	
	7.50	0.93 ± 0.02	0.48 ± 0.02	147.96 ± 0.67	
	10.00	0.90 ± 0.03	0.57 ± 0.07	164.54 ± 0.98	
	KCl	0.50	1.77 ± 0.04	0.51 ± 0.05	157.49 ± 0.63
		1.00	1.41 ± 0.02	0.34 ± 0.03	144.62 ± 0.54
2.00		1.31 ± 0.03	0.27 ± 0.04	132.47 ± 0.87	
3.00		1.25 ± 0.02	0.47 ± 0.02	138.96 ± 1.06	
4.00		1.16 ± 0.02	0.55 ± 0.05	142.78 ± 1.02	
5.00		1.12 ± 0.01	0.59 ± 0.04	155.04 ± 0.99	
7.50		1.10 ± 0.02	0.61 ± 0.05	149.63 ± 0.86	
10.00		1.08 ± 0.03	0.63 ± 0.04	164.28 ± 0.85	

^a Ref [22]; ^b Ref [23].

$a = 0.0079 \pm 0.0003$, $b = 0.0543 \pm 0.0079$ and $c = 76.04 \pm 4.23$ ($R^2 = 0.996$)

For all the other salts, values of coefficients are:

NH₄Cl: $a = 0.0075 \pm 0.0005$, $b = 0.0474 \pm 0.0143$, $c = 95.27 \pm 7.63$ ($R^2 = 0.995$)

KCl: $a = 0.0084 \pm 0.0003$, $b = -0.2692 \pm 0.0634$, $c = 84.68 \pm 6.89$ ($R^2 = 0.991$)

MgCl₂: $a = 0.0076 \pm 0.0002$, $b = 1.7321 \pm 0.3194$, $c = 2184 \pm 43$ ($R^2 = 0.989$)

In the presence of salts with different cations, the CMC of SDBS decreases as the salt is added in aqueous SDBS solution which is due to the compression of the electric double layer at the interface due to electrostatic interactions. This facilitates the adsorption of surfactant molecules at the interface and thus reduces the CMC.^{21–23} Among the various salts, the CMC of SDBS

follows the sequence: MgCl₂ < KCl < NH₄Cl < NaCl as shown in Figure 2. It can be explained by considering the ratio of valence (Z) and van der Waals radius (R), Z/R . The cations having larger Z/R value are more hydrated and water structure makers.¹⁶ These ions tend to decrease the CMC values more as they possess a strong ability to salt out the hydrophobic groups of the surfactant from the aqueous phase. Since Z/R value is maximum (9.62) for divalent Mg²⁺ ions and lowest (4.35) for Na⁺ among monovalent ions,¹³ the decrease in CMC is highest in the presence of MgCl₂ and the least for NaCl.

3.2 Influence of different salts on α

The decrease or increase in α value (Table 1) for different salts varies with the type and composition of the salts. In case of Na⁺ ions, α value initially increases

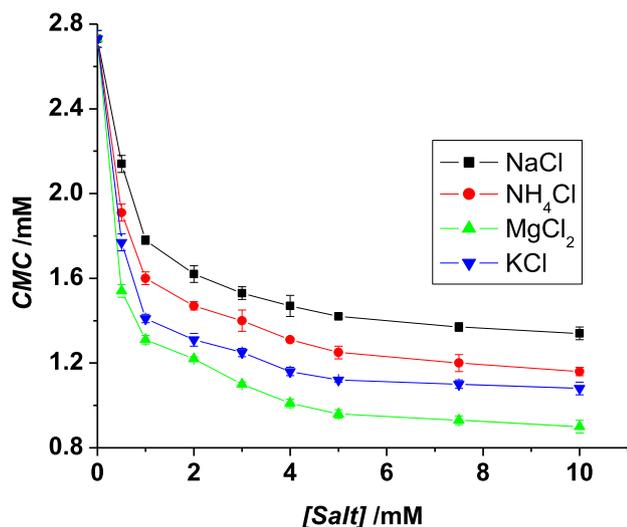


Figure 2. Effect of salts on the CMC of SDBS. Drawn lines are guide to the eye.

with [salt] and then starts to decrease after reaching a maximum (Figure 3). The initial increase in α at low concentrations is either due to the micellar growth or increase in the screening of the charge.²⁵ This increase in the micellar size would result in a decrease in the charge density causing the release of the counter ions.²⁴ At higher concentrations (7.5 and 10 mM) of Na^+ ion, the α values decrease due to binding of a large number of counter ions to the micellar surface, resulting in a decrease in α value²⁶ in aqueous solutions possessing high ionic strength. In case of NH_4^+ ions, the α value decreases initially; after reaching a minimum, the value starts to increase and then decreases at higher [salt] similar to that for Na^+ ion. However, for Mg^{2+} and K^+ ions, the effective ionic charge is the highest among the studied cations. Therefore, the decrease in α value is sharp at low salt concentrations. Hence, the CMC of SDBS decreases drastically in the presence of Mg^{2+} and K^+ in comparison to NH_4^+ and Na^+ ions.

The α values decrease at lower concentrations of the studied salts (except NaCl) which can be attributed to the strong interactions of SDBS micelles with Mg^{2+} , K^+ and NH_4^+ in comparison to Na^+ ion. Among the studied cations, the net ionic charge is more in the case of Mg^{2+} ions even though the hydration of these ions is the highest. Thus, the decrease in α value among the cations follow the sequence: $\text{Mg}^{2+} > \text{K}^+ > \text{NH}_4^+$. In case of NaCl, the α value increases at lower concentrations because Na^+ ions are more screened as compared to K^+ and NH_4^+ ions as the hydration of Na^+ ions is more than that K^+ and NH_4^+ ions. Further, the CMC values of SDBS decrease drastically in the presence of Mg^{2+} and

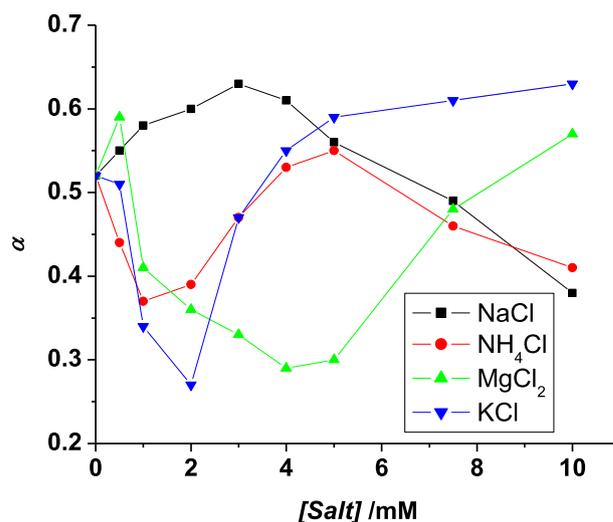


Figure 3. Effect of salts on α values for SDBS. Lines drawn are guide to the eye.

K^+ in comparison to that of Na^+ and NH_4^+ ions (Figure 2) as the effective ionic charge on Mg^{2+} and K^+ ions are the highest among the studies of electrolytes.

3.3 Influence of various salts on derived parameters

The aggregation number (N) for SDBS has been calculated as,²⁷

$$N = K ([Counterion^+]_{aq})^\gamma \quad (4)$$

Where, $[Counterion^+]_{aq}$ represents the composition of counter ions (in moles) supplied by both added salts and SDBS. K and γ are constants that depend upon the length of the alkyl chain²⁸ and their values for SDBS are 158 and 0.16, respectively. The term $[Counterion^+]_{aq}$ is given by the following relationship:²⁷

$$[Counterion^+]_{aq} = \alpha [SDBS] + (1 - \alpha) CMC + [salt] \quad (5)$$

The values of N thus obtained are presented in Table 2 and agree well with those obtained using fluorescence measurements.²³ Since the electrostatic repulsions between the head groups decrease in the presence of salt¹¹, therefore, the N values increase with [salt] and this increase is more in the presence of Na^+ ions. The radius of the micelle, r , has been obtained from,²⁹

$$r = \left[\frac{3}{4\pi} (27.4 + 26.9n_c) N \right]^{1/3} \quad (6)$$

Here, the number of carbon atoms per hydrocarbon chain of the surfactant is exhibited by n_c . Since the values of r are directly proportional to N , hence, these values also increase with [salt]. From the knowledge of

Table 2. Aggregation number (N), surface area per head group (A_o), packing parameter (P_m), volume per surfactant molecule (V_p), hydrodynamic diameter (D_h) and zeta potential (ζ) for SDBS at 298.15 K.

Salt	[Salt]/mM	N	$A_o/\text{\AA}^2$	P_m	$V_p/\text{\AA}^3$	D_h/nm	ζ/mV	
NaCl	0.00	57 (61) ^c	78 (74) ^c	0.31	557.23	2.9	-82.1	
	0.50	65	71	0.33	458.62	3.2	-64.4	
	1.00	69	65	0.35	423.58	3.3	-68.2	
	2.00	72	61	0.36	411.25	4.3	-55.1	
	3.00	73	59	0.38	372.66	6.6	-50.4	
	4.00	76	54	0.40	352.84	8.7	-45.6	
	5.00	79	52	0.41	338.91	12.9	-47.9	
	7.50	71	49	0.37	296.55	18.3	-43.8	
	10.00	59	44	0.36	244.68	27.8	-43.2	
	NH ₄ Cl	0.50	59	75	0.32	476.57	3.2	-63.1
1.00		61	71	0.32	463.09	3.5	-68.5	
2.00		64	68	0.33	452.16	4.7	-51.2	
3.00		68	65	0.35	439.27	6.7	-48.8	
4.00		71	62	0.34	416.85	11.0	-45.2	
5.00		73	60	0.35	391.62	17.5	-46.5	
7.50		68	56	0.36	365.08	26.1	-42.1	
10.00		61	52	0.38	331.26	34.9	-41.6	
MgCl ₂		0.50	58	75	0.33	523.68	3.8	-55.3
		1.00	59	70	0.34	504.24	4.2	-47.2
	2.00	62	68	0.34	473.82	4.7	-41.4	
	3.00	64	66	0.35	462.51	5.9	-38.7	
	4.00	65	65	0.34	437.66	8.6	-33.2	
	5.00	63	62	0.37	392.87	19.9	-32.1	
	7.50	58	58	0.35	374.62	25.4	-31.7	
	10.00	62	55	0.37	335.84	45.6	-28.2	
	KCl	0.50	59	74	0.34	489.52	3.5	-58.4
		1.00	62	72	0.33	471.06	3.9	-47.6
2.00		64	71	0.35	445.38	4.9	-48.9	
3.00		63	65	0.36	411.27	6.8	-43.2	
4.00		68	60	0.34	381.66	10.2	-40.3	
5.00		71	56	0.35	348.57	18.6	-38.2	
7.50		77	52	0.35	326.54	28.7	-36.6	
10.00		84	47	0.37	286.35	39.4	-35.1	

^c Ref [23].

r , the micellar surface area per head group (A_o) may be found using,³⁰

$$A_o = \frac{3v}{r} \quad (7)$$

v is the volume corresponding to the hydrophobic chain of the micelle³¹ and is given by $v = 27.4 + 26.9n_c \text{\AA}^3$. A_o value in aqueous solution of SDBS is understandably higher (Table 2) than the value of minimum area (A_{\min}) reported in the literature.²³ These values decrease with increase in [salt] due to close packing of the adsorbed molecules as a consequence of better screening of the head groups in presence of salts.¹¹ The packing parameter, P_m , has been calculated as,

$$P_m = \frac{v}{A_o l} \quad (8)$$

$l = 1.5 + 1.265n_c$ and represents the hydrophobic chain length. For spherical micelles, $P_m = 0.33$; cylindrical micelles $P_m = 0.50$ and for disks and bilayers, $P_m = 1$. Generally, P_m is calculated by taking A_{\min} value as an approximation to A_o value. But, we have estimated the accurate values of P_m using A_o . From Table 2, it can be seen that P_m value increases slightly with [salt] indicating formation of larger micelles. The volume of each molecule of surfactant in the shell of the micelle (V_p) has been obtained as,²⁸

$$V_p = \frac{4\pi}{3N} [(r + 5)^3 - r^3] \quad (9)$$

The calculated value of V_p (Table 2) decreases with the increase in [salt] and the decrease is more in the case of NaCl. The limiting ionic equivalent conductance for SDBS micelles (λ_o^-) has also been evaluated using

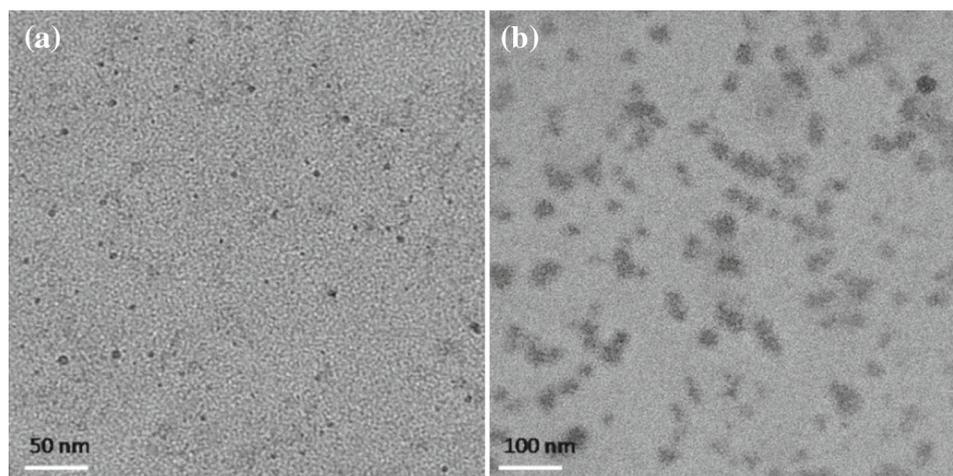


Figure 4. TEM image of SDBS micelles in (Left) aqueous solution; (Right) 10 mM NaCl solution.

Stokes-Einstein equation.²⁵

$$\lambda_o^- = \frac{N\alpha e_o F}{6\pi\eta r} \quad (10)$$

Here, η is the viscosity of water; F is the Faraday constant and e_o is the charge. In the presence of the studied salts, the value of λ_o^- for SDBS micelles changes differently with [salt] as the charge ($N\alpha$) depends upon the kind and [salt] added. Hence, λ_o^- decreases and increases in the same range of [salt] as α .

3.4 Hydrodynamic diameter and Zeta potential

DLS experiments were performed to measure the hydrodynamic diameter (D_h) of SDBS micelles in aqueous solutions of different salts. D_h for SDBS micelles (without salt) is 2.9 nm which is lower than 3.4 nm obtained previously for SDS micelles³² although both the surfactants have the same dodecyl chain in their monomers. It can be understood based on the fact that SDBS monomer has one extra benzene ring. As a consequence, the head groups cannot come close beyond a certain limit due to the repulsive interactions of the π -electron cloud of the benzene ring present at the micellar surface. Therefore, the hydrocarbon chain of SDBS acquires folded confirmation and hence D_h of SDBS is expected to be less than that of SDS.

The increase in D_h of SDBS micelles with [salt] is due to an increase in electrostatic repulsion between the head groups as shown clearly in the TEM image (Figure 4). However, the increase in D_h is not much at lower [salt] but beyond 5 mM, the size of SDBS aggregate increases suddenly, which is an indication of the shape change as confirmed by P_m values. The magnitude of zeta potential (ζ) provides a measure of the stability of the colloidal system. The negative values of ζ is due to the charge

on SDBS micelles. The magnitude of ζ decreases with increasing [salt] as a result of a decrease in thickness of the electric double layer (Gouy-Chapman layer). Both D_h and ζ values supplement the observations from conductivity measurements.

4. Conclusions

A precise and iterative method has been used to study the influence of some salts on the CMC and fraction of counter ion dissociation of SDBS in aqueous solutions. All the studied salts decrease the CMC of SDBS according to the sequence: $\text{MgCl}_2 < \text{KCl} < \text{NH}_4\text{Cl} < \text{NaCl}$ which has been attributed to the difference in their charge to radius ratio. The aggregation number increases whereas surface area per head group decreases with [salt]. DLS studies exhibit an increase in D_h upon addition of salts which is further confirmed by TEM images. The ζ values were negative whose magnitude decreases in the presence of salts due to the decrease in the thickness of the electric double layer. The limiting equivalent conductance of SDBS micelles is more in the presence of Na^+ and K^+ in comparison to NH_4^+ and Mg^{2+} ions.

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