International Journal of Global Science Research **ISSN: 2348-8344 (Online)**
Vol. 10, Issue. 1, April 2023, pp. 2068-2078 **DOI: 10.26540/ijgsr.v10.i1.2023.239**
***Available Online* at www.ijgsr.com**

Research paper

Effect of Cation Valence on the Micellar Properties of Sodium bis (2-ethyl hexyl) Sulphosuccinate (AOT) in Aqueous Medium

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Received: 15/03/2023
Revised: 19/03/2023
Accepted: 23/03/2023

Abstract: From tensiometric measurements, critical micelle concentration (CMC), maximum surface excess concentration (Γ_{max}) and minimum area per molecule (A_{min}) of Sodium bis (2-ethyl hexyl) sulphosuccinate (AOT) at air-liquid interface in aqueous solutions with *or* without an added electrolyte of varying cation valence have been determined, at 288.15, 293.15 and 298.15 K. Whereas, the CMC of AOT+H_2O solution invariably decreases on adding an electrolyte and is further lowered when an electrolyte of higher cation valence is mixed, on the contrary, it is raised upon increasing the solution temperature. The process of micellization in the bulk solution and adsorption at air-liquid interface are both favored by exothermic enthalpy as well as entropy gain. The CMC lowering effect upon mixing an electrolyte was found in the order: ($AlCl_3$)>($CaCl_2$)>(NaCl). Thus, mixing of electrolyte with higher cation valence would be of more advantage in lowering the CMC of the surfactant. This can be exploited for improving the efficiency of the surfactant as a detergent, for managing the problem of oil spill and for more efficient extraction of petroleum oil in the tertiary process.

Introduction:

Studies on interactions of inorganic ions on the physico-chemical properties of surfactants in aqueous solution provide an insight into their actual performance in agriculture, textile, detergency, (Demissie, H. *et. al.*, Akhlaghi, N. *et. al.*) pharmaceuticals, cosmetic, solubilization, enhanced oil recovery, (Naskar, B. *et. al.*, Belhaj, A.F. *et. al.*) emulsifying power, wetting capacity and in metallurgical processes (Ram Partap *et. al.*). Although several reports have appeared on different physico-chemical properties of surfactants in aqueous solutions (Dominguez, H. *et. al.*, Sukul, D. *et. al.*, Watry, M. R. *et. al.*, Ram Partap *et. al.*, Ghosh S. *et. al.*, Dutta, P. *et. al.*, Yekeen, N. *et. al.*), yet to the best of our knowledge no report exists, at present, on the effects of cation valencies on surface and thermodynamic properties of sodium bis (2-ethyl hexyl) sulphosuccinate (AOT) in aqueous medium. We report, here, critical micelle concentrations (CMC), surface pressure at CMC (π_{cmc}), surface excess concentration (Γ_{max}), minimum area per molecule (A_{min}) at the air–liquid interface, and thermodynamic parameters of micellization, adsorption and transfer of AOT in aqueous medium. Effects of cation valency of some electrolytes on the above physico-chemical properties of the studied surfactant at 288.15, 293.15 and 298.15 K have been presented.

Materials and Methods:

Chemicals:

Sodium Chloride (NaCl, MW: 58.44 g mol^{-1}); Calcium Chloride ($CaCl_2$, MW: 110.98 g mol^{-1}), Aluminum Chloride ($AlCl_3$, MW: 133.34 g mol^{-1}) and Sodium bis (2-ethyl hexyl) Sulphosuccinate ($C_{20}H_{38}NaO_7S$; MW: 445.57 g mol^{-1}) were from SD Fine Chemicals. Molecular structure of Sodium bis (2-ethyl hexyl) Sulphosuccinate (AOT) is shown in Fig. 1. Doubly distilled water (specific conductance 2.0 x 10^{-6} S cm^{-1}) was used for preparing various aqueous AOT solutions.

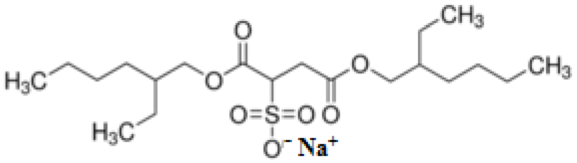

Figure:1. Molecular structure of Sodium bis (2-ethyl hexyl) Sulphosuccinate (AOT).

Method:

Surface tensions of Sodium bis (2-ethyl hexyl) Sulphosuccinate (AOT) aqueous solutions, with or without added an electrolyte (NaCl, $CaCl_2$ or $AlCl_3$) were measured by drop-weight method using a modified stalagmometer, described elsewhere (Jain, D.V.S. *et. al.*). It was calibrated using standard liquids including *n*-hexane, benzene, carbon tetrachloride, acetophenone and water. Surface tensions were measured over a wide range of AOT concentrations and at temperatures 288.15, 293.15 and 298.15 K. using a thermostatic bath (Tempstar, Model KW 201 A) that ensured temperature control within ± 0.01 K. The reproducibility of measured surface tension values was within ± 0.2 mNm $^{-1}$. A digital conductivity meter (Model E.I. 601 E) was employed for conductance measurements of surfactant solutions.

Results and Discussion:

Critical Micelle Concentration

Critical micelle concentration (CMC) of AOT solutions with or without added electrolyte at studied temperatures were obtained from the break-point on their respective surface tension versus log [AOT] plots. The CMCs of AOT aqueous solutions without an added electrolyte were also obtained from the break point on specific conductivity versus [AOT] plots and break point on viscosity versus [AOT] plots. Typically, plots of surface tension versus log [AOT], specific conductance versus [AOT] and viscosity verses [AOT] for AOT+H_2O system at 288.15, 293.15 and 298.15 K are presented in Figs. 2 to 4, respectively.

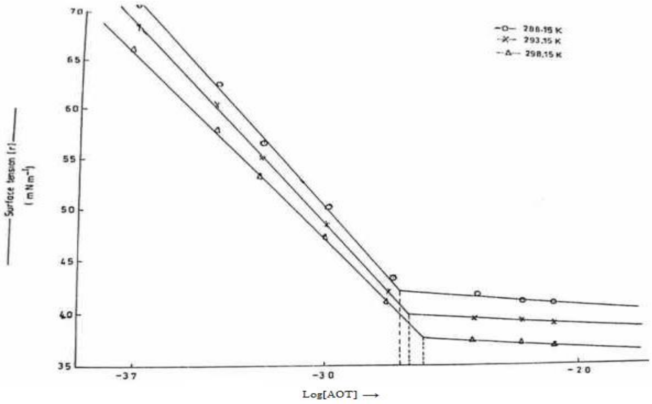

Figure: 2. Plot of surface tension (γ /mNm^{-1}) as a function of Log [AOT] at 288.15, 293.15 and 298.15 K for aqueous AOT solutions.

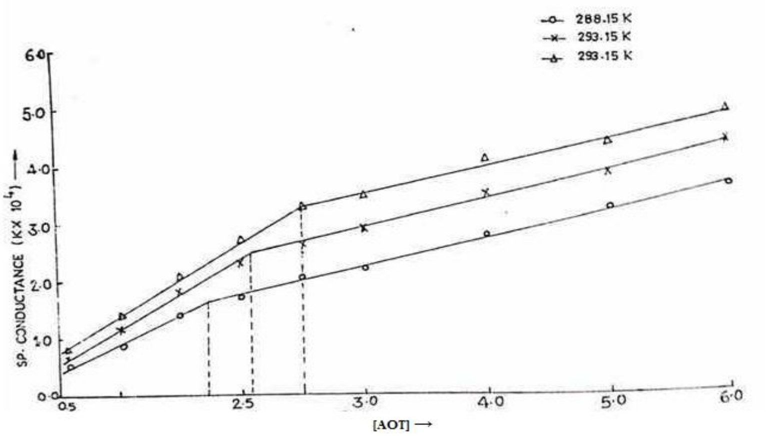

Figure: 3. Plots of specific conductivity (k)x10^4 (Scm^{-1}) as a function of [AOT] at 288.15, 293.15 and 298.15 for aqueous AOT solutions.

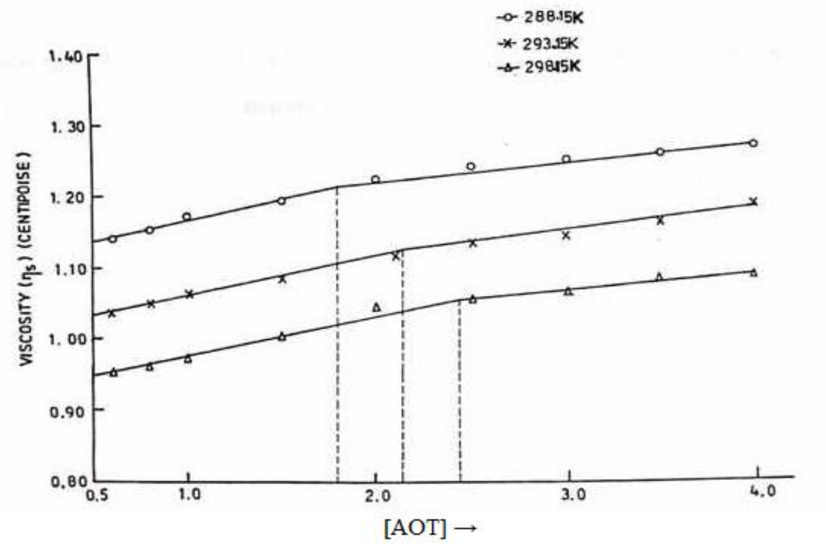

Figure: 4. Plots of specific viscosity (η_s) as a function of [AOT] at 288.15, 293.15 and 298.15 for aqueous AOT solutions.

The values of CMC, thus obtained, for the studied systems are presented in Table-1.

Table: 1. Critical micelle concentration (CMC), surface excess concentration (Γ_{max}), minimum area per molecule (A_{min}) and surface pressure at CMC (π_{cmc}) for AOT+ water system with or wihout an added electrolyte of varying cationic valency.

System	Temp (K)	CMC x 10^3 (mol dm^{-3})	Γ_{max} x 10^{10} (mol cm^{-2})	A_{min} (nm^2)	π_{cmc} (mNm^{-1})
AOT+H_2O	288.15	1.82(1.84* 1.84**)	2,68	6.19	31.7
	293.15	2.12(2.14* 2.14**)	2.55	6.51	32.4
	298.15	2.42(2.44* 2.50**)	2.42	6.86	35.4
AOT+H_2O+0.025M NaCl	288.15	1.50	2.22	7.48	42.5
	293.15	1.70	1.87	8.88	42.8
	298.15	1.90	1.54	10.78	43.4
AOT+H_2O+0.050M NaCl	288.15	1.33	2.10	7.91	46.5
	293.15	1.53	1.75	9.48	47.4
	298.15	1.70	1.50	11.07	48.6
AOT+H_2O+0.075M NaCl	288.15	1.16	2.04	8.14	47.3
	293.15	1.36	1.60	10.38	48.8
	298.15	1.56	1.46	11.37	49.2
AOT+H_2O+0.025M $CaCl_2$	288.15	1.30	1.53	10.85	45.7
	293.15	1.46	1.41	11.78	46.9
	298.15	1.60	1.32	12.58	48.2
AOT+H_2O+0.050M $CaCl_2$	288.15	1.18	1.47	11.29	47.3
	293.15	1.33	1.39	11.94	47.4
	298.15	1.48	1.30	12.77	47.6
AOT+H_2O+0.075M $CaCl_2$	288.15	1.04	1.40	11.86	48.2
	293.15	1.18	1.34	12.39	48.4
	298.15	1.34	1.28	12.86	48.5
AOT+H_2O+0.025M $AlCl_3$	288.15	0.96	1.41	11.78	47.3
	293.15	1.12	1.23	13.49	47.6
	298.15	1.26	1.10	15.09	47.9
AOT+H_2O+0.050M $AlCl_3$	288.15	0.84	1.34	12.39	47.6
	293.15	1.02	1.19	13.95	48.1
	298.15	1.12	1.01	16.44	48.2
AOT+H_2O+0.075M $AlCl_3$	288.15	0.70	1.20	13.84	48.3
	293.15	0.89	1.08	15.37	48.6
	298.15	0.98	0.96	17.29	48.8

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*CMC values from viscosity measurements
**CMC values from conductance measurements

The observed CMC for AOT+H_2O at 298.15K agrees well with the values reported in literature (Hek, S.R. *et. al.*, Chatterjee, A. *et. al.*, Mukhim, T. *et. al.*, Ram Partap *et. al.,* Akhter, M. S.). The CMC values for the studied systems invariably increase with increasing temperature. This may be due to the diminished hydration sphere around the AOT's ionic head groups, caused by enhanced thermal agitation at higher temperature, resulting in their more ion-ion repulsions and thus shifting surfactant monomers ⇌ micelle equilibrium in favor of the monomers leading to higher CMC (Jakubowska, A., Ma, X. *et. al.*).The CMC of AOT solutions decreases with an added electrolyte due to (a) enhanced dielectric constant of aqueous medium which promotes early formation of normal micelles and (b) increased charge density of AOT counter-ion causing a diminished inter-molecular head-group repulsion thus enhancing the surfactant monomers' association ability (Patra, N. *et. al.,* Liu, Z. *et. al.,* Ram Partap *et. al.*). A still higher concentration of an added electrolyte in AOT solution results in contraction of the electrical double layer around the micelle leading to the reduction in its CMC value (Tennouya, L. *et. al.*).

The effect of electrolyte's cation valency on decreasing CMC of AOT was found in the order: (trivalent Al^{+3}) > (divalent Ca^{+2})>(monovalent Na^{+}).

Maximum Surface Excess Concentration

Maximum surface excess concentration (Γ_{max}) of the surfactant (AOT) at the air-liquid interface was obtained using Gibb's adsorption equation (Rosen, M.J. *et. al.*)

$$\Gamma_{max} = -\,(1/2.303\,nRT)\,(d\gamma/\,d\log C)_T \quad (1)$$

where 'n' is the number of particles released per surfactant molecule in aqueous solution (for the ionic surfactant AOT, $n = 2$); R = gas constant (8.3145 J. $mol^{-1}K^{-1}$) and C is the surfactant molar concentration. The term $(d\gamma\,/d\log C)_T$ is given by the slope of the plot for surface tension (γ) versus log C , at constant T, prior to micelle formation (*i.e* before CMC). The values of $\Gamma_{\mathbf{max}}$ for the studied systems, at varying concentrations of an added electrolyte and at different temperatures, are also presented in Table 1. The $\Gamma_{\mathbf{max}}$ values decrease with increasing temperature which may be due to the enhanced thermal agitation at higher temperature causing a partial shifting of surfactant monomers from the air-liquid interface to the bulk (Sharma, V. K. *et. al.*). The $\Gamma_{\mathbf{max}}$ values further decrease upon mixing an electrolyte in AOT solution owing to the displacement of a fraction of surfactant molecules at the air-liquid interface by electrolyte ions.

Minimum Area per Molecule

Minimum area per molecule ($A_{\mathbf{min}}$) at the liquid–air interface has been calculated using the equation (Rosen, M.J. *et. al.*)

$$A_{min} = 10^{14}/\,(N.\,\Gamma_{max}) \qquad (2)$$

Where, 'N' is the Avogadro's Number ($6.022\text{x}10^{23}$ mol^{-1}). The values of A_{min}, presented in Table 1, show the positive dependence on temperature as well as on the amount of added electrolyte. It may be because at a higher temperature, due to the expansion of liquid, the available free space per molecule is increased allowing it to stretch further and an added electrolyte causes a partial disruption of water-structure, enabling further relaxation of the surfactant monomers.

Surface Pressure at CMC

Surface pressure at CMC (π_{cmc}), an index of the surface tension reduction at CMC, was obtained using the relation (Rosen, M.J. *et. al.*)

$\pi_{cmc} = \gamma_0 - \gamma_{cmc}$ (3)

Where, γ_0 = surface tension of pure water and γ_{cmc} = surface tension of aqueous surfactant (AOT) solution at CMC. The calculated π_{cmc} values (Table-1) increase with increasing temperature and are further enhanced upon mixing an electrolyte in AOT solution. It may be because the higher temperature and the addition of an electrolyte both cause weakening of hydrogen-bonded water-structure in the bulk thus cumulatively decrease the intermolecular interaction in aqueous medium resulting in lowering of the surface tension at CMC and hence the observed higher $\pi_{cmc.}$

Thermodynamic Properties of Micellization

Standard Gibb's free energy of micellization (ΔG^0_{mic}) for AOT in an aqueous solution without an added electrolyte, was calculated using the equation (Berry, B. W. *et. al.*)**:**

$\Delta G^0_{mic} = (2-\beta)\, RT \ln X_{cmc}$ (4)

Where, β is the fraction of micellar charge un-neutralized by the counter ions and was determined from the ratio of post micellar and pre micellar slopes of specific conductance versus [surfactant] plot for AOT solutions without added an electrolyte. However, for aqueous surfactant (AOT) solutions, containing an electrolyte, the value of β was taken as one, since the counter ion effect was suppressed by the added electrolyte. R are T are gas constant and temperature in Kelvin, respectively, X_{cmc} represents the mole fraction of surfactant at CMC. The standard state for surfactant is the solvated surfactant monomer at unit mole fraction referred to infinitely dilute solution, and for micelle, the micelle itself is considered as its standard state.

Further, the standard entropy of micellization (ΔS^0_{mic}) and enthalpy of micellization (ΔH^0_{mic}) were obtained using equations (6) and (7), respectively (Rosen, M.J. *et. al.*).

$\Delta S^0_{mic} = -d(\Delta G^0_{mic})/\, dT$ (5)

$\Delta H^0_{mic} = \Delta G^0_{mic} + T\Delta S^0_{mic}$ (6)

The above thermodynamic properties of micellization, presented in Table-2, suggest that the micelle forming process in aqueous media is favored both by entropy gain as well as by exothermic enthalpy change. The ΔG^0_{mic} values of AOT in pure water as well as in electrolyte solutions are negative suggesting the feasibility of AOT micelle formation in aqueous media. Upon raising the temperature, ΔG^0_{mic} decreases (become more negative) for AOT aqueous solutions owing to the enhanced entropy gain by disruption of water structure higher temperature (Mc. Donald). The observed further decrease of ΔG^0_{mic} upon mixing an electrolyte in AOT solution, may be due to even larger entropy increase caused by a more severe disruption of hydrogen bonded water structure by the ions of the added electrolyte (Rosen, M.J. & Aronson, S.).

Table 2- Thermodynamic parameters of micellization / adsorption and transfer for aqueous solutions of AOT with or without an added electrolyte of varying cationic valency.

System	Temp./K	$-\Delta G^0_{mic}$/$-\Delta G^0_{ad}$ / $-\Delta G^0_{tr}$ (kJ mol^{-1})	$-\Delta H^0_{mic}$ /$-\Delta H^0_{ad}$ / ΔH^0_{tr} (kJ mol^{-1})	ΔS^0_{mic} / ΔS^0_{ad} /ΔS^0_{tr} (kJK^{-1}mol^{-1})
AOT+ H_2O	288.15	27.88 / 29.52 / -	21.68 / 26.96 / -	0.172 / 0.196 / -
	293.15	28.74 / 30.50 / -	22.27 / 28.13 / -	0.174 / 0.200 / -
	298.15	29.62 / 31.52 / -	22.85 / 29.30 / -	0.176 / 0.204 / -
AOT+ H_2O + 0.025M NaCl	288.15	25.23 / 27.63 / 0.49	-	-
	293.15	25.32 / 27.99 / 0.53	17.62 / 6.31 / 2.69	0.026/0.074/ 0.011
	298.15	25.48 / 28.37 / 0.60	-	-
AOT+ H_2O + 0.050M NaCl	288.15	25.48 /27.76 / 0.75	-	-
	293.15	25.58 / 28.27/ 0.79	17.37 / 5.44 / 3.02	0.028 / 0.115 / .013
	298.15	25.76 / 28.91/ 0.88	-	-
AOT+ H_2O + 0.075M NaCl	288.15	25.77 / 27.93 / 1.04	-	-
	293.15	25.88 / 28.47 / 1.09	16.79/ 10.50 / 3.60	0.031 / 0.133 / 0.016
	298.15	26.08 / 29.26 / 1.20	-	-
AOT+ H_2O + 0.025M $CaCl_2$	288.15	25.53 / 28.83 / 0.80	-	-
	293.15	25.69 / 29.02 / 0.90	14.85 / 11.15 / 5.55	0.037 / 0.061 / 0.022
	298.15	25.90 / 29.44 / 1.02	-	-
AOT+ H_2O + 0.050M $CaCl_2$	288.15	25.76 / 28.98 / 1.03	-	-
	293.15	25.92 / 29.34 / 1.13	15.96 / 06.47 / 4.44	0.034 / 0.078 / 0.019
	298.15	26.10 / 29.76 / 1.22	-	-
AOT+ H_2O + 0.075M $CaCl_2$	288.15	26.07 / 29.28 / 1.34	-	-
	293.15	26.21 / 29.66 / 1.42	18.30 / 05.91 / 2.09	0.027 / 0.081 / 0.012
	298.15	26.34 / 30.09 / 1.46	-	-
AOT+ H_2O + 0.025M $AlCl_3$	288.15	26.26 / 29.61 / 1.53	-	-
	293.15	26.35 / 30.22 / 1.55	19.31 / 6.11 / 1.09	0.024 / 0.124 / 0.009
	298.15	26.50 / 30.85 / 1.62	-	-
AOT+ H_2O + 0.050M $AlCl_3$	288.15	26.46 / 30.22 / 1.73	-	-
	293.15	26.57 / 30.87 / 1.78	17.19 / 8.12/3.20	0.032 / 0.133 / 0.017
	298.15	26.78 / 31.55/ 1.90	-	-
AOT+ H_2O + 0.075M $AlCl_3$	288.15	26.82 / 30.74 / 2.10	-	-
	293.15	26.96 / 31.44 / 2.17	15.53 /10.72 / 4.57	0.039 / 0.144 / 0.023
	298.15	27.21 / 32.18 / 2.33	-	-

Standard entropy of micellization (ΔS^0_{mic}) are positive for AOT+H_2O solutions and increase with increasing temperature due to the dispersion of water structure as described above. Whereas, mixing of NaCl or $AlCl_3$ to an aqueous AOT solution results in increase of ΔS^0_{mic} value, reverse is true when $CaCl_3$ is added. This may be explained in terms of lower tendency of water structure breaking by less hydrated Ca^{+2} ions compared to more hydrated Na^+ or Al^{+3} ions in aqueous medium. Standard enthalpies of micellization, (ΔH^0_{mic}) are exothermic for AOT+H_2O solutions and the exothermicity is further enhanced upon raising the temperature. It may be because at higher temperature more free (unstructured) water molecules are available for ion hydration. However, upon mixing an electrolyte in aqueous AOT solution the ΔH^0_{mic} increases (become less negative) due to dominance of exothermic ion hydration effect over the endothermic water structure disruption effect by the added electrolyte.

Thermodynamic Parameters of Adsorption

Thermodynamic parameters of adsorption (viz . ΔG^0_{ad}, ΔH^0_{ad} and ΔS^0_{ad}) of AOT monomers at liquid-air interface have been evaluated using the relations[30]:

$$\Delta G^0_{ad} = RT \ln X_{cmc} - 6.023 \times 10^{-1} \pi_{cmc}. A_{min} \quad (8)$$

$$\Delta S^0_{ad} = - d(\Delta G^0_{ad}) / dT \quad (9)$$

$$\Delta H^0_{ad} = \Delta G^0_{ad} + T \Delta S^0_{ad} \quad (10)$$

Where, R, T, X_{cmc} π_{cmc} and A_{min} are as defined above. The values of ΔG^0_{ad}, ΔH^0_{ad} and ΔS^0_{ad} thus obtained are also presented in Table 2. In case of all the studied systems, the values of ΔG^0_{ad} are invariably lower (more exothermic) compared to corresponding ΔG^0_{mic}, suggesting that the process of adsorption of AOT monomers at the air-liquid interface is thermodynamically more feasible compared to their aggregation to form micelles. The higher entropies of adsorption (ΔS^0_{ad}) in comparison to ΔS^0_{mic} may be due to more degree of freedom of the surfactant monomers at the liquid-air interface compared to the cramped interior of micelles (Wertz, D. H. *et. al.,* Rosen, M.J.). The standard enthalpy of adsorption (ΔH^0_{ad}) for AOT+H_2O solutions are more exothermic compared to ΔH^0_{mic} at the studied temperatures which may be attributed to larger entropy gain by higher degree of freedom of surfactant monomers at the liquid surface with their head group pointed toward the surface and their hydrophobic tail pointing away from the liquid surface. However, the same is reversed when an electrolyte is added to AOT solution. This could be due to more intensive ion hydration in bulk solution than at the air-liquid interface.

Thermodynamic parameters of transfer:

Standard thermodynamic parameters of transfer (viz. $\mathbf{\Delta G^0_{tr}}$, ΔH^0_{tr} and $\mathbf{\Delta S^0_{tr}}$) from binary (AOT+H_2O) to ternary (AOT+H_2O+electrolyte) solution were evaluated using following relations (Rosen, M.J. and Aronson, S.) and are recorded in Tables 2.

$$\Delta G^0_{tr} = \Delta G^0_{mic(ter)} - \Delta G^0_{mic(bin)} \quad (11)$$

Where, $\Delta G^0_{mic(ter)}$ and $\Delta G^0_{mic(bin)}$ are the standard free energy of micellization of the ternary and the binary solutions, respectively.

$$\Delta S^0_{tr} = - d(\Delta G^0_{tr}) / dT \quad (12)$$

$$\Delta H^0_{tr} = \Delta G^0_{tr} + T\Delta S^0_{tr} \quad (13)$$

The values of ΔG^0_{tr} , ΔH^0_{tr} and ΔS^0_{tr} thus obtained are also included in Table-2. The standard Gibb’s free energy of transfer (ΔG^0_{tr}) for the studied systems are negative indicating the feasibility of the transfer of micelles from a binary surfactant solutions to the corresponding ternary solution due to water structure disruption due to the added electrolyte which facilitates such transfer. The ΔG^0_{tr} values decrease at higher temperature due to breaking of hydrogen-bonded structure of water, at higher temperature, causing the entropy gain. The increase of an electrolyte concentration as well as the higher cation valence of the added electrolyte, due to their respective water cluster breaking effect, cumulatively lower the ΔG^0_{tr} values. Further, though endothermic ΔH^0_{tr} opposes the transfer of AOT from pure water to electrolyte solution, yet the same is predominately favoured by entropy gain.

Conclusion:

Effects of some electrolytes on the physico-chemical and thermodynamic properties of an anionic surfactant, sodium bis (2-ethyl hexyl) Sulphosuccinate (AOT), in aqueous medium have been studied by surface tension measurements. It is found that mixing of an electrolyte lowers the critical micelle concentration (CMC) of the surfactant which even further lowers with increasing cationic valency of the electrolyte. These findings would be beneficial for improving the efficiency of AOT in its use as laundry detergent, solubilization of organic material in water, froth floatation in metallurgical process, recovery of petroleum in tertiary processes and managing the oil spill problem.

Graphical Abstract

Tensiometric study has revealed that an electrolyte added to Sodium bis (2-ethyl hexyl) Sulphosuccinate (AOT) surfactant aqueous solution lowers its critical micelle concentration (CMC) which further decreases with increasing cationic valency of the electrolyte. It would be beneficial for improving the efficiency of AOT as a laundry detergent, managing the problem of oil spill and in its several applications.

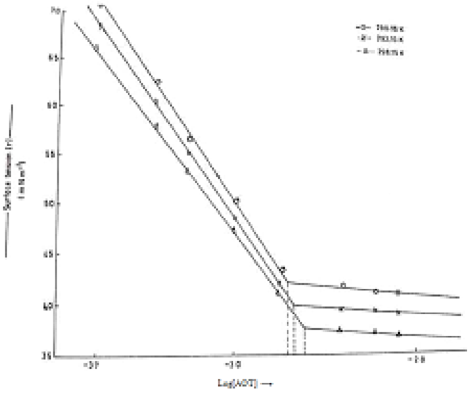

Figure: Plots of surface tension (γ /mNm^{-1}) as a function of Log [AOT] at 288.15, 293.15 and 298.15 K for aqueous AOT solutions.

Acknowledgement:
The authors (SB & RP) are grateful to the Principal, Govt. P G College Hisar, for kindly allowing basic research facilities and providing moral support for carrying out this research work.

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