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Viscometric Studies of Ion Solvation of Some Alkali Metal Salts in 2-Aminoethanol + *N*,*N*-Dimethylacetamide Binary Mixtures at 298.15 K and 308.15 K

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Densities (ρ) and viscosities (η) of lithium perchlorate ($LiClO_4$), sodium perchlorate ($NaClO_4$), potassium thiocyanate (KSCN), tetrabutylammonium thiocyanate (Bu_4NSCN), tetrabutylammonium tetraphenylborate (Bu_4NBPh_4) and tetrabutylammonium perchlorate (Bu_4NClO_4) were measured in binary mixed solutions of 2-aminoethanol (AE) and *N*,*N*-dimethylacetamide (DMA) containing 25, 50, 60, 80 and 100 mol % DMA in the concentration range (3-500) $\times 10^{-4}$ mol kg^{-1} at 298.15 K and 308.15 K temperatures. The density and viscosity data were analyzed using the Jones-Dole equation in the form $\eta_r = 1 + A(C^{1/2}) + BC$ for unassociated electrolytes and viscosity A and B-coefficients were obtained from the plots of of $\eta_r - 1/C^{1/2}$ *versus* $C^{1/2}$. The A-coefficients obtained from the analysis of Jones-Dole equation are positive as well as negative. The positive values indicate the solvation of ions in solutions of binary mixtures (AE + DMA) especially in 50 and 60 mol% DMA. The viscosity *B*-coefficients of all these electrolytes were positive and large in case of cations and were split into their respective ionic ($B_\pm$) coefficient values using a method reported by Gill and Sharma. The viscosity B-coefficients values were increasing with the increase increasing DMA composition in the binary mixed solvent system of AE + DMA but decreasing with increasing temperature from 298.15 K to 308.15 K. The obtained results indicate about the preferential solvation of ions *i.e.* Li^+, Na^+, K^+, Bu_4N^+ and Ph_4B^- ions and poor solvation of ClO_4^- and SCN^- ions. The results also revealed that these interactions get weaker with increase in temperature. The resulting B-coefficients and its dependence of temperature provide useful information regarding changes in the solvent structure.

Keywords: 2-Aminoethanol, *N*,*N*-Dimethylacetamide, Viscosity B-coefficients, Solvation.

INTRODUCTION

Solvation chemistry plays an important role in the study of electrolytic solutions of alkali metals in binary mixtures of both aqueous and non-aqueous solvents with other organic solvents. Electrolytic studies of metal salts in different solvents and their binary mixtures have various applications in metallurgical industries, solvent extraction, high power batteries, reaction kinetics, supercapacitors, electroplating, reaction mechanisms and in the areas of biological and pharmaceutical sciences, *etc*. [1-4]. This study area is becoming remarkably interesting in the present as well as in future scenario as the demand of secondary energy storage devices based on lithium and sodium ions is increasing day-by-day. It has vast applications in various fields of convenient electrical machines due to their high power and output voltage. The behaviour of alkali metal salts in binary mixed solvents provide an idea of solvation characteristics of their ions as well as about their interaction in the solvent whether of ion-ion or ion-solvent type. Several solvation studies related to the alkali metal salts and tetrabutylammonium salts [5,6] were employing their use as 1:1 electrolytes and these were mostly solvated by purely electrostatic ion-dipole interactions. Ion solvation studies in organic binary mixed solvents as well as in aqueous medium are also very common [7-9]. The studies revealed that metal and metal ions have been playing a big role in binary mixed solvents in order for these to be used as better electrolytic system for lithium-ion batteries (LIBs) and sodium-ion batteries (NIBs).

The ionic associations of salt solutions depends on the type of solvation of its ions [10]. For this purpose, the viscosity and the relative permittivity were taken into consideration to determine the extent of the ion-associations and the solvent-

solvent interactions. Thus, in recent past decade, ample studies were performed on viscosity and density in various polar and non-polar as well as in aqueous mixed organic solvents to scrutinize the behavior of ion-ion and ion-solvent interactions.

One of the solvents *N*,*N*-dimethylacetamide (DMA), is a dipolar aprotic solvent and has a high dielectric constant (37.8) and dissolves all salts to a greater extent. 2-Aminoethanol (AE) on the other hand, is a polar aprotic in nature which has an industrial and biological importance but it does not dissolve some of salts [10,11]. Therefore, it is mainly used as a solvent in the non-aqueous electrochemistry [12,13]. The present work on the alkali metal salts, provides an insight into the viscometric behaviour of the electrolytes *i.e.* lithium perchlorate ($LiClO_4$), sodium perchlorate ($NaClO_4$), potassium thiocyanate (KSCN), tetrabutylammonium thiocyanate (Bu_4NSCN), tetrabutylammonium tetraphenylborate (Bu_4NBPh_4) and tetrabutylammonium perchlorate (Bu_4NClO_4) in the concentration range 0.005-0.2 mol kg^{-1} in 25, 50, 60, 80 and 100 mol% of DMA in AE + DMA binary mixtures at 298.15 K and 308.15 K. The salts Bu_4NClO_4 and Bu_4NBPh_4 were used as reference electrolytes. The solubility of $LiClO_4$ and Bu_4NBPh_4 is not appreciable in pure AE [11], which makes it difficult to evaluate density and viscosity of both the electrolytes in pure AE. Therefore, the densities and viscosities all the salts were studied in pure DMA as well as in its binary mixtures with AE.

EXPERIMENTAL

2-Aminoethanol (99.5% (GC grade, TCI Japan) and *N*,*N*-dimethylacetamide (99.5%, (GC grade, TCI Japan) were used without further purification. The densities of 2-aminoethanol were found to be 1.019 g cm^{-3} at 298.15 K and 1.00443 g cm^{-3} at 308.15 K [10-12]. The densities of *N*,*N*-dimethylacetamide were found to be 0.9360 g cm^{-3} at 298.15 K and 0.9264 g cm^{-3} at 308.15 K. The viscosities of 2-aminoethanol were found to be 18.96 cP at 298.15 K and 12.267 cP at 308.15 K, while the viscosities of *N*,*N*-dimethylacetamide were found to be 0.9279 cP at 298.15 K and 0.8182 cP at 308.15 K agree well with literature values [13,14]. Lithium perchlorate (Hi-Media, AR grade) was twice purified by recrystallization in acetonitrile as reported method [15]. Sodium perchlorate (98.5%, Rankem) was purified by recrystallization in acetone whereas potassium thiocyanate (Alfa Aesar, ACS Grade 99%) and tetrabutylammonium thiocyanate (> 98% AR grade, TCI Japan) were used without further purification. The procedure for preparation of reference electrolytes *i.e.* tetrabutylammonium perchlorate (Bu_4NClO_4) and tetrabutylammonium tetraphenylborate (Bu_4NBPh_4) was followed as per the reported method [16].

Densities of the solvents (AE and DMA), their binary solvent mixtures (AE + DMA) and their salt solutions have been measured with a Borosil glass made pyknometer of capacity 10 cm^3 with a stem of 0.05 cm^3 divisions and was calibrated by double distilled water and some reference solvents such as acetone, DMA, hexane, *etc*. at desired experimental temperatures. The weight of the empty pyknometer with a Teflon cap was first taken at desired experimental temperatures (298.15 K and 308.15 K) by using an A & D company limited electronic balance (Japan, Model GR-202) with precision of ± 0.01mg. The sample was introduced in to the pyknometer through a hypodermic syringe with a precaution that no air bubble was produced. Then the mouth of filled pyknometer was closed with teflon cap and weighed. The pyknometer was immersed in a thermostat maintained at experimental temperature and the level of the solution was observed in the pycnometer, which was utilized in the calculation of density data. Measurements were performed thrice for every solution at different temperatures. The reproducible results were obtained with standard minimum error of $\pm 2 \times 10^{-5}$ g cm^{-3}. The approximate values of densities of pure solvents were found very close to the literature values with uncertainty in the value as ± 0.0001g cm^{-3}. The viscosities of the solution samples were measured by means of Ubbelohde suspended level viscometer with fine capillary tube suspended vertically in refrigerated water bath thermostat maintained at the experimental temperatures with an uncertainty of ± 0.01 ºC. The cleaning of the used apparatus for both the measurements to obtained viscosities and densities data was done by first using distilled water, then ethanol and finally with acetone to get quick dryness for accurate results.

The viscosity values were evaluated with the help of the given equation [17]:

$$\eta = \rho\left(Kt - \frac{L}{t}\right) \tag{1}$$

where K and L are the constants for a given viscometer. Their evaluated values are K = 0.048 cm^2/s^2 and L = 3.12 cm^2.

RESULTS AND DISCUSSION

The physico-chemical properties *i.e.* densities (ρ), viscosities (η) and dielectric constants (ε) of AE, DMA and their binary mixtures containing 25, 50, 60, 80, and 100 mol% DMA in AE and their salt solutions at 298.15 K and 308.15 K are shown in Table-1. The values of density and viscosity data were assessed by applying the experimental relationships and determined at 298.15 K and 308.15 K. These values of the solvents agreed well to the literature values whereas the values of dielectric constants were taken from literature [11].

Furthermore, it is essential to realize the intermolecular interactions among the used solvent mixture compositions to acknowledge the dependency on viscosity and density [1-3].

The viscosities of $LiClO_4$, $NaClO_4$, KSCN, Bu_4NSCN, Bu_4NBPh_4 and Bu_4NClO_4 were analyzed using Jones-Dole equation [18-20] in binary mixed solutions of 2-aminoethanol (AE) and *N*,*N*-dimethylacetamide (DMA) [19,20].

The Jones-Dole equation in the form

$$\eta_r = 1 + A(C^{1/2}) + BC \tag{2}$$

where A is the ionic atmosphere coefficient and B is the effective ion size parameter that also contains contributions from ion-ion and ion-solvent effects. Here, η_o is the viscosity of pure solvent while η is the viscosity of the used salt of molar concentration (c) is dissolved therein solution. The coefficients are the measure of ion-ion interaction and can be calculated theoretically from limiting molar conductance data and thermo-physical parameters of the solvent using Falkenhagen-Vernon equation [21]. For calculation of A_η, the limiting molar conductance

TABLE-1
RELATIVE PERMITTIVITIES (ε), DENSITIES (ρ) (g cm^{-3}) AND VISCOSITIES (η) (cP) OF BINARY SOLVENT MIXTURES (AE + DMA) AT EXPERIMENTAL TEMPERATURES

Mol% DMA	Physical property	298.15 K	308.15 K
0	ε	34.20	33.05
	ρ	1.0117	1.00443
	η	18.960	12.267
25	ε	36.89	35.30
	ρ	0.96476	0.95985
	η	7.735	4.105
50	ε	38.52	37.29
	ρ	0.93955	0.93913
	η	2.021	1.584
60	ε	39.68	37.45
	ρ	0.93106	0.93069
	η	1.752	1.465
80	ε	40.81	39.47
	ρ	0.91393	0.91336
	η	1.152	0.955
100	ε	43.00	42.50
	ρ	0.93604	0.92643
	η	0.9279	0.8182

(Λ^o), of the electrolyte in the used solvent system and the limiting ionic conductances, λ_1^o and λ_2^o are required. At this stage, the data of λ_1^o and λ_2^o are not available in actual concentration for the present solvent system. Therefore, it is not possible to determine the A_η values of used electrolytes in pure DMA and binary mixtures of AE + DMA.

$$A_\eta = \frac{0.2577\lambda_o}{\eta_o(\varepsilon_o T)^{1/2}\lambda_1^o\lambda_2^o}\left[1 - 0.6863\left(\frac{\lambda_1^o - \lambda_2^o}{\Lambda_o}\right)\right] \quad (3)$$

The values of A and B coefficients for all used salts were determined by the ion-size and by ion-solvent and solvent-solvent interactions. Some workers [22] have also observed that the experimental A and B coefficients values evaluated from the plot of

$$\psi = \frac{\eta - \eta_o}{\eta_o C^{1/2}} = \frac{\eta}{\eta_o} - \frac{1}{C^{1/2}} \quad (4)$$

or $\eta_r - 1/C^{1/2}$ *versus* $C^{1/2}$ in non-aqueous solvents were different from the A_η values and found to be straight line at all the concentrations of the used salts. Some cases have negative A values but these values have no significance [4,23]. In 50 and 60 mol% DMA, the A values are positive show substantial solvation of all used electrolytes in it. This data indicates that the strong ion-ion interactions. These A values were given in Table-2.

TABLE-2
A-COEFFICIENTS (dm$^{3/2}$ mol$^{-1/2}$) OF THE JONES-DOLE EQUATION VALUES FOR USED SALTS IN AE + DMA MIXTURES AT 298.15 K AND 308.15 K

Salts	Temp. (K)	Mol% DMA				
		25	50	60	80	100
		$10^2 A$	$10^2 A$	$10^2 A$	$10^2 A$	$10^2 A$
Bu_4NBPh_4	298.15	-0.53	4.40	1.76	-0.08	1.85
	308.15	-0.47	4.19	0.57	0.35	0.62
Bu_4NClO_4	298.15	-0.15	1.19	1.71	-0.26	1.41
	308.15	-0.17	1.18	1.55	-0.28	-0.006
$LiClO_4$	298.15	-0.43	4.03	3.29	-0.75	-3.41
	308.15	-0.43	1.62	1.09	-0.76	-3.29
$NaClO_4$	298.15	-0.65	0.35	1.48	2.69	-0.42
	308.15	-0.58	0.35	1.24	2.40	-0.44
KSCN	298.15	-0.47	0.34	3.01	1.71	-0.36
	308.15	-0.46	0.36	2.64	0.72	-1.33
Bu_4NSCN	298.15	-0.54	0.66	1.39	-2.95	-0.68
	308.15	-0.64	0.59	1.06	-2.84	0.69

2.2 [14] and 2.38 [13], 1.48 [14]

This means that the B-coefficients provide the information about the ion-solvent interactions. The calculated densities and viscosities data were used in evaluating the

$$\psi = \frac{\eta - \eta_o}{\eta_o C^{1/2}} = \frac{\eta}{\eta_o} - \frac{1}{C^{1/2}}$$

or $\eta_r - 1/C^{1/2}$ *versus* $C^{1/2}$ of $LiClO_4$, $NaClO_4$, KSCN and Bu_4NSCN in the AE + DMA binary mixtures containing 25, 50, 60, 80 and 100 mol% DMA as a function of concentration at 298.15 K and 303.15 K.

Ionic B_+ and B_- coefficients: According to the studies of Gill *et al.* [24,25] confirmed that the ionic radii of Bu_4N^+ and Ph_4B^- are almost constant in organic non-aqueous solutions and their binary mixed solvent system and equal to 5.00 and 5.35 Å, respectively using for calculating the indivi-dual ionic $B_\pm$ coefficients. The ionic B_+ and B_- coefficients (Table-3) have been obtained by splitting of viscosity coefficients of electrolytes on the method suggested by Gill *et al.* [14] presented in Table-3. The separation of viscosity coefficients of electrolytes into individual ion contributions provided by Gill & Sharma method [14,26,27], which is based on the Bu_4NBPh_4 assumption, according to the following eqns.:

TABLE-3
B-COEFFICIENTS (dm^3 mol^{-1}) OF THE JONES-DOLE EQUATION FOR USED SALTS IN AE + DMA MIXTURES AT 298.15 AND 308.15 K

Salts	Mol% DMA									
	298.15 K					308.15 K				
	25	50	60	80	100	25	50	60	80	100
Bu_4NBPh_4	1.30	1.36	1.39	1.53	2.25	1.22	1.35	1.37	1.45	2.22
Bu_4NClO_4	1.03	1.06	1.08	1.12	1.50	0.96	0.98	0.99	1.06	1.41
$LiClO_4$	0.61	0.78	0.88	1.03	1.13	0.59	0.73	0.83	1.01	1.08
$NaClO_4$	1.00	1.11	1.13	1.18	1.42	0.92	1.02	1.04	1.15	1.35
KSCN	0.64	0.87	1.10	1.26	1.87	0.62	0.94	1.07	1.19	1.40
Bu_4NSCN	1.05	1.09	1.12	1.21	1.56	1.02	1.06	1.09	1.18	1.54

2.41 [14], 2.311 [13]

TABLE-4
IONIC $B_{\pm}$ ((dm^3 mol^{-1}) COEFFICIENTS FOR SOME IONS IN AE + DMA MIXTURES AT 298.15 AND 308.15 K

Ions	Mol% DMA									
	298.15 K					308.15 K				
	25	50	60	80	100	25	50	60	80	100
Bu_4N^+	0.584	0.611	0.625	0.688	1.011	0.548	0.607	0.615	0.652	0.998
Ph_4B^-	0.716	0.749	0.765	0.842	1.239	0.672	0.743	0.755	0.798	1.222
ClO_4^-	0.446	0.449	0.425	0.432	0.489	0.414	0.373	0.374	0.408	0.412
SCN^-	0.466	0.479	0.495	0.522	0.549	0.472	0.453	0.475	0.528	0.542
Li^+	0.179	0.349	0.458	0.598	0.641	0.184	0.352	0.470	0.602	0.668
Na^+	0.534	0.661	0.705	0.748	0.931	0.509	0.647	0.680	0.742	0.938
K^+	0.178	0.390	0.675	0.738	0.859	0.147	0.442	0.595	0.662	0.858

$$\frac{B_{Ph_4B^-}}{B_{Bu_4N^+}} = \frac{r^3_{Ph_4B^-}}{r^3_{Bu_4N^+}} = \left(\frac{(5.35)^3}{(5.00)^3}\right) \qquad (7)$$

$$B_{Ph_4B^-} + B_{Bu_4N^+} = B(Bu_4NBPh_4) \qquad (8)$$

From eqns. 7 and 8, the viscosity B-coefficients of the experimental salt solutions in binary mixtures of evaluated were split into the ionic contributions (B_+) of the individual ions and are displayed in Table-4. The $B_{\pm}$ values were positive and large for Bu_4N^+, Li^+, Na^+, K^+ and Ph_4B^- in DMA rich region of binary mixtures. The B_+ and B_- coefficient values for Bu_4N^+ and Ph_4B^-, however, show an increase in DMA concentration region in binary mixtures of AE + DMA, indicates the preferential solvation by DMA in AE + DMA binary mixtures. This data confirmed about the general nature of non-aqueous solvents [8,28-31]. The SCN^- and ClO_4^- anions have constant B_- values in AE + DMA binary mixtures as well as in pure DMA, indi-cating their poor solvation. This behavior was confirmed in dipolar aprotic solvents [31-33]. The ionic B_- values of Ph_4B^- were determined, which were used to calculate the ionic contri-bution of ClO_4^- and Bu_4N^+ ions (Table-4). The viscosity B-co-efficients of all splitted ions were found to decrease with increase in temperature. It is evident that the solvation of ions in DMA rich region decreases with the increase in temperature in the binary mixtures of AE + DMA.

It has been observed from the order that the solvation of Li^+ ion is less in comparison to Na^+ and K^+ ion. Similar approach was observed by Mukherjee *et al*. [31] and Das *et al*. [34], in some other non-aqueous solvents. It is due to greater solvation interactions by DMA because of the availability of lone pair of electrons on N-atom in DMA which it donates to Li^+, Na^+ and K^+ ions to form coordinate covalent bond (especially with Li^+ and Na^+ ions). This order can be explained on the basis of charge density, as the ions with high charge density interact strongly with the solvent molecules, and decrease the viscous flow of the solvent [35].

Conclusion

The values of viscosity B-coefficients for the electrolytes and ions are large and positive, which shows the preferential solvation of Li^+, Na^+ and K^+, Bu_4N^+ and Ph_4B^- ions by DMA in AE + DMA binary mixtures while poor solvation of SCN^- and ClO_4^- ions in the same solutions, which might be due to bulkiness of ions. The viscosity B-coefficients of the used electrolytes in the binary mixtures of AE + DMA, decrease with the increase in temperature from 298.15 K and 308.15 K. The A-values were positive in 50 and 60 mol% DMA show strong solvation of all used electrolytes in it and indicates the substantial/preferential solvation of electrolytes in binary mixtures of AE + DMA. The reliable values of B-coefficients and its ions studies to the viscosity suggests about the strong ion-solvent and ion-ion interactions but these interactions were decrease with the increase in temperature.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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