

Research Article

Thermophysical, Volumetric, and Excess Properties of Aqueous Solutions of 1-Hexyl-3-methyl Imidazolium Bromide at 298.15 K and 0.1 MPa

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Room temperature ionic liquids (RTILs) are the environment-friendly alternatives for organic volatile solvents in a host of synthetic, catalytic, and electrochemical applications. These are also being used for various R&D works in nuclear fuel cycle research such as the recovery and purification of nuclides of interest from spent nuclear fuel matrices. In this work, density, refractive index (n_D^{25}) at sodium *D* line, and thermodynamic water activity of the aqueous solution of 1-hexyl-3-methyl imidazolium bromide were determined in dilute aqueous solutions at 298.15 K. These results were used to calculate the apparent molal volumes of each solute over various concentration ranges. The measurements were performed as per ASTM procedures. Vapour pressure was derived using water activity values. Information on excess properties and structural interaction was also reported.

1. Introduction

Room temperature ionic liquids are considered as young chemicals having variety a of applications in all types of areas in chemical industry due to their unique properties. They are referred to as designer solvents as we can design them for different reactions by changing the cation and anion. They can act as both polar and nonpolar solvents which minimizes the use of lot of chemicals making our environment neat and clean and thus referred to as “green solvent” [1]. Researchers have found that ionic liquids are more than a solvent and have several applications in electrochemistry, biological media, catalysis, organic synthesis, and so on [2–7]. Low vapour pressure, thermal stability, high thermal conductivity, large electrochemical window, and nonflammability are some of their properties which make them an innovative solvent. As a solvent, ionic liquids possesses several advantages over conventional organic solvents making it environmentally compatible [8]. The potential advantage of ionic liquids depends upon their thermophysical properties like density, refractive index, activity, vapour pressure, conductivities, viscosities, and so forth. Various research groups have studied

the thermophysical properties of various ionic liquids to extract important information about them [9–16]. RTILs are extensively used for the extraction of metal ions in solvent extraction [17–19]. In nuclear industry these are used as green solvent for the extraction of various fission products in liquid-liquid extraction [20–22]. Currently, variants of 1,3 dialkyl imidazolium salt are being tried in processing of materials. In this paper one of such 1,3-dialkyl imidazolium salts that is, 1-hexyl-3-methyl imidazolium bromide (hereafter referred as [HMIm]Br), is studied extensively whose properties are not available yet. Here, apparent molal volume, refractive index, water activity, vapour pressure, and other derived properties of aqueous solutions of [HMIm]Br at 298.15 K and 0.1 MPa are reported.

2. Experimental Work

2.1. General Procedure. ASTM Grade-1 water as per ASTM D-1193 [23] with a resistivity of 18.2 M Ω ·cm at 298.15 K and TOC < 15 ppb from a MILLIPORE Simplicity system was used in the experiments for reagent/solution preparation and

as water standard. Water for density standard was drawn from the water purifier immediately before the experiments and it was degassed in an ultrasonic bath (TEC-110 H) at 35 KHz for 60 seconds. Solutes are weighed in a precision Shimadzu AUW220D balance (220 g, 0.01 mg resolution). When not in use, the solutions were kept in air-tight closed condition in a JULABO SW-22 Shaker Bath at 298.15 ± 0.1 K. JULABO SW-22 Shaker Bath was coupled to JULABO FP-40 chiller for cooling. To ensure the ripple-free power supply to all the equipments, AC supply was regulated with an uninterrupted power supply (APC), set at high sensitivity. The ambient conditions were monitored with a Cole-Parmer hygrometer (0.1%) consisting of a thermometer (0.1°C) and pressure sensor (0.1 kPa). The compound [HMIm]Br (M/S *io-li-tec*, GmbH, structure shown in Figure 1) was examined by IR spectra shown in Figure 2 (3079.8 cm^{-1} , 2931.5 cm^{-1} , 2860.2 cm^{-1} , 1642.9 cm^{-1} , 1465.7 cm^{-1} , 1166.8 cm^{-1} , and 1627.8 cm^{-1}) and used without further purification.

2.2. Measurement of Density and Refractive Index. For density and refractive index measurement, a precise vibrating-tube densitometer (Anton Paar DMA-5000), coupled with Anton Paar ABBEMAT RXA-156 refractometer and a 30-station autosampler SP-3 m, was used. The densitometer and refractometer had Peltier thermostats for maintaining the desired sample temperature. Density measurements were made in complete confirmation of ASTM-D4052 [24].

2.3. Measurement of Water Activity. Water activity was measured by a thermodynamic water activity meter (NOVASINA AW-Master) with a built-in thermoelectric Peltier element module for maintaining temperature of sample. Samples were loaded in polystyrene cups and placed into the activity meter. The activity meter was standardized using a two-step procedure. In the first step, purified and degassed Millipore water was used as a standard ($a_w = 1.000$). In the second step, five immobilized electrolyte solutions, in sealed tablets supplied by M/S Novasina, were used as standards. These solutions were ~25% LiCl ($a_w = 0.113$), ~25% $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ($a_w = 0.328$), ~25% NaCl ($a_w = 0.753$), 25% $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ($a_w = 0.901$), and ~20% K_2SO_4 ($a_w = 0.973$). The uncertainty estimated was ± 0.1 K for temperature and ± 0.003 for a_w values.

The aqueous solutions of [HMIm]Br were made (~ 0.0005 m to 0.04 m) by the authors. The density, refractive index, water activity, and other derived properties for 1-hexyl-3-methyl imidazolium bromide solutions were determined and reported.

3. Results and Discussion

3.1. Theory. Partial molar volume is an extensive property and can be expressed by

$$\bar{V}_i = \left(\frac{\partial V}{\partial n_i} \right)_{T, P, n_{j \neq i}} \quad (1)$$

For electrolytes, this partial molar quantity is composition dependent. In such cases apparent molal volume of

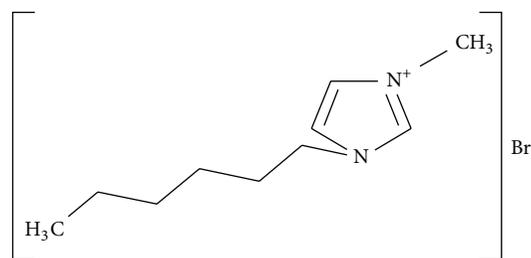


FIGURE 1: Structure of [HMIm]Br.

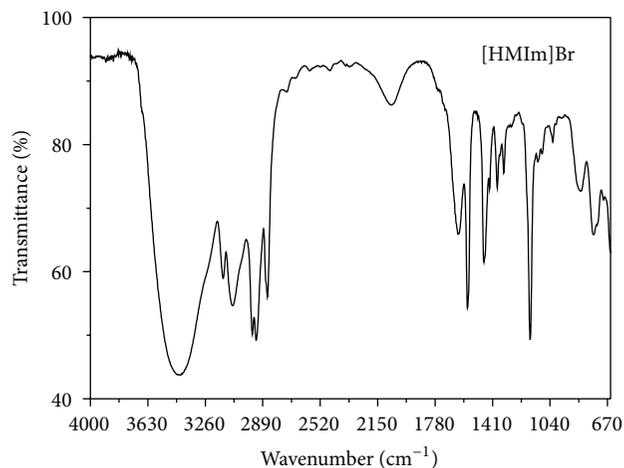


FIGURE 2: IR data of aqueous solutions of pure [HMIm]Br.

component 2 in a solution (solvent is defined as component 1) is given by

$$V_{2,\phi} = \frac{V - n_1 V_1^0}{n_2} \quad (2)$$

The apparent molar volume of a solute (designated as 2) in a solvent in a solution may be written as

$$V_{2,\phi} = \frac{1000}{m\rho\rho_0} (\rho_0 - \rho) + \frac{M_2}{\rho} \quad (3)$$

where $V_{2,\phi}$ is the apparent molar volume, m is the molality, M_2 is the molecular weight of the solute, and ρ_0 and ρ are the densities of water and of the solution, respectively.

There are several empirical correlations to correlate the apparent molar volume of a solute in the solution with the solute concentration. The following equation lists the correlation proposed by Masson [25]:

$$V_{2,\phi}^{298.15\text{K}} = V_{2,\phi}^0 + a_v \sqrt{m} \quad (4)$$

where $V_{2,\phi}^0$ is the limiting apparent molal volume, m is the molality, and a_v is an empirical constant determined from experimental results. The slope of the plot between $V_{2,\phi}$ and " m " will give the value of a_v and the intercept will give the value of $V_{2,\phi}^0$.

TABLE 1: Density values of aqueous solution of [HMIm]Br at 298.15 K.

Conc. (m)	Expt. density (g/cm ³)	Pred. density (g/cm ³)	Residuals* (g/cm ³)	Deviations**%
0	0.9970	0.9970	0	0
0.00158	0.9971	0.9971	-0.0000	-0.0023
0.0028	0.9971	0.9971	-0.0000	-0.0009
0.0034	0.9971	0.9971	-0.0000	-0.0009
0.0043	0.9973	0.9973	-0.0000	-0.0014
0.0054	0.9973	0.9973	-0.0000	-0.0008
0.0079	0.9974	0.9974	-0.0000	-0.0001
0.0082	0.9974	0.9974	0.0000	0.0006
0.0101	0.9975	0.9975	0.0000	0.0003
0.0155	0.9978	0.9978	0.0000	0.0008
0.0231	0.9987	0.9987	0.0000	0.0004
0.0351	0.9991	0.9991	-0.0000	-0.0004

* Calculated as $\rho_{\text{pred}} - \rho_{\text{expt}}$.

** Calculated as $100 * (\rho_{\text{pred}} - \rho_{\text{expt}}) / \rho_{\text{expt}}$.

The average deviation for the data of Table 1 = $0.9063E - 003\%$.

The standard deviation for the data of Table 1 = $-0.3958E - 003\%$.

On equating (3) and (4) and rearranging, following form of equation for density of solution is obtained:

$$\frac{\rho}{\rho_0} = 1.00 + a_0 \frac{m}{m_0} + b_0 \left(\frac{m}{m_0} \right)^{1.5}, \quad (5)$$

where

$$a_0 = \frac{M_2 - \rho_0 \times V_{2,\phi}^0}{10^3}, \quad (6)$$

$$b_0 = -\frac{a_0}{10^3}.$$

Similarly, the experimentally determined refractive index values of aqueous solutions of ionic liquid were correlated to the following equation:

$$nD = nD_w + am + bm^2, \quad (7)$$

where nD is the predicted refractive index of solution and nD_w is the refractive index of water used in solution preparation. m is molality of ionic liquid in water. a and b are adjustable coefficients.

Excess molar volume of solution is a thermodynamic property and is defined as the amount of volume which is in excess to that of an ideal solution at the same conditions of composition, temperature, and pressure and is given as

$$V_m^E = V_m - V_m^{\text{ideal}}, \quad (8)$$

where V_m and V_m^{ideal} are the molar volumes of solution and pure components, respectively. Excess molar volumes of aqueous [HMIm]Br solution were calculated using (9) which was obtained after rearranging (8) using mole fractions, molar masses, and densities to represent the volumes of solution and pure components:

$$V_m^E / \text{cm}^3 \cdot \text{mol}^{-1} = x_1 M_1 \left(\frac{1}{\rho} - \frac{1}{\rho_0} \right) + x_2 M_2 \left(\frac{1}{\rho} - \frac{1}{\rho_1} \right), \quad (9)$$

where V_m^E is the excess molar volume; M_1 and M_2 are molar masses of water and [HMIm]Br, respectively; x_2 is the mole fraction of [HMIm]Br; ρ is the density of aqueous [HMIm]Br solutions; and ρ_0 and ρ_1 are densities of Millipore water and pure [HMIm]Br, respectively.

The excess molar volumes were correlated with a Redlich-Kister-type equation by least-squares fittings given by

$$V_m^E / \text{cm}^3 \cdot \text{mol}^{-1} = x_1 x_2 \sum_{i=0}^n A_i (2x_i - 1)^i, \quad (10)$$

where A_i are the adjustable coefficients.

Excess refractive index was also calculated in the same manner using the Redlich-Kister polynomial.

3.2. Correlation of Density of Aqueous Solutions [HMIm]Br. The experimental data (listed in Table 1) could be correlated by the following equation:

$$\frac{\rho}{\rho_0} = 1.00 + 0.049 \frac{m}{m_0} - 0.009 \left(\frac{m}{m_0} \right)^{1.5}. \quad (11)$$

The density of ASTM Grade-I water at 298.15 K was taken as 0.997043 g/cm^3 [24]. The mean deviation and standard deviations were $0.9063 \times 10^{-3}\%$ and $-0.3958 \times 10^{-3}\%$, respectively. A good match between experimental and predicted values was observed. The deviation in density values was given in Figure 3.

3.3. Correlation of Apparent Molal Volume. The apparent molal volumes for [HMIm]Br in aqueous solution at 298.15 were calculated with (3) (listed in Table 2) and correlated with (4). The first few values were not used in regression. The following correlation is obtained:

$$V_{2,\phi}^{298.15\text{K}} = 195.39 + 31.74 \sqrt{m}. \quad (12)$$

The correlated and experimentally determined values are shown in Figure 4.

TABLE 2: Apparent molal volume values of [HMIm]Br as a function of concentration.

Molality (mol · kg ⁻¹)	$V_{2,\phi}$ (cm ³ · mol ⁻¹)
0.0016	193.7778
0.0043	196.1672
0.0054	197.9337
0.0079	199.5758
0.0082	200.3581
0.0101	200.008
0.0155	200.5182
0.0351	200.6666
0.0434	200.6378

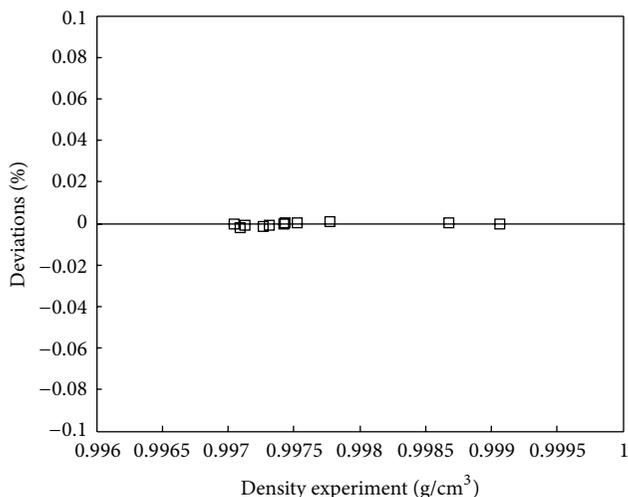


FIGURE 3: Plot of deviations for density of aqueous solutions of [HMIm]Br in water at 298.15 K and 0.1 MPa.

3.4. *Correlation of Refractive Index.* Experimental refractive index values have been shown in Table 3. The correlated equation is given as

$$nD = 1.3324 + 0.0386m - 0.0021m^2. \quad (13)$$

The variation of refractive index for aqueous solutions of [HMIm]Br at 298.15 K and 0.1 MPa with concentration is given in Figure 5.

Experimental water activity values have been shown in Table 3. From determined water activity values, vapour pressures were estimated as

$$P = a_w \times P^0, \quad (14)$$

where $P^0 = 3.1686$ kPa at 298.15 K. Figures 6 and 7 show variation of water activity and vapour pressure for aqueous solutions of [HMIm]Br at 298.15 K and 0.1 MPa with concentration.

At infinity dilution there will be no solute-solute interaction. Here the positive values of $V_{2,\phi}^0$ are a result of strong ion-solvent interaction. In this case as the solvent volume

TABLE 3: Experimental data for refractive index, water activity, and vapour pressure of aqueous solution of [HMIm]Br.

Conc. m	Refractive index nD^{25}	Water activity a_w	Vapour pressure kPa
0	1.332428	1.000	3.1686
0.0005	1.332456	0.999	3.1654
0.0015	1.332483	0.998	3.1623
0.00158	1.332487	0.998	3.1623
0.0028	1.332528	0.997	3.1591
0.0034	1.332553	0.996	3.1559
0.0043	1.332593	0.996	3.1559
0.0054	1.332638	0.995	3.1528
0.0079	1.332737	0.994	3.1496
0.0082	1.332752	0.994	3.1496
0.0101	1.332832	0.993	3.1464
0.0155	1.333033	0.991	3.1401
0.0231	1.333308	0.990	3.1369
0.0351	1.333775	0.987	3.1274
0.0434	1.334107	0.986	3.1242

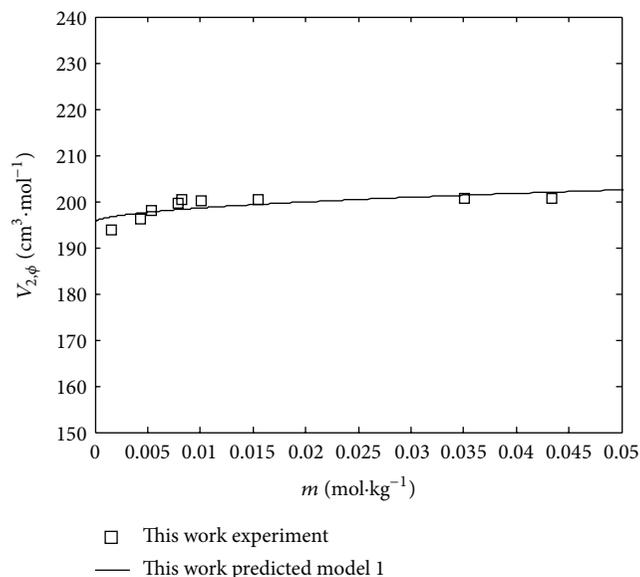


FIGURE 4: Apparent molal volume of [HMIm]Br in water at 298.15 K.

is decreasing due to strong ion-solvent interaction, the apparent molal volume is increasing with the addition of the electrolyte.

Excess volume is a resultant contribution of physical, chemical, and structural effects. The physical contribution involving nonspecific interactions between the real species of mixture contribute a positive value to V^E . The chemical interactions involving charge-transfer-type forces result in a decrease in volume and contribute a negative value to V^E . The structural contributions are mostly negative which arises from changes of free volume and other various effects. In this experiment, excess molar volume is found to be negative as is common in completely miscible solvents. The negative excess

TABLE 4: Excess volume and excess refractive index values of aqueous solution of [HMIm]Br.

x_2	V^E	nD^E
9.8724E-06	-2.162E-05	2.49E-05
2.7139E-05	-5.875E-05	6.83E-05
2.8499E-05	-6.164E-05	7.18E-05
5.1205E-05	-0.000109	0.000129
6.0814E-05	-0.0001286	0.000153
7.7629E-05	-0.0001622	0.000195
9.832E-05	-0.0002025	0.000246
0.00014169	-0.0002827	0.000353
0.00014689	-0.0002919	0.000366
0.00018296	-0.0003539	0.000455
0.0002798	-0.0005011	0.00069
0.00041635	-0.0006616	0.001014
0.00063257	-0.0008034	0.001512
0.00078072	-0.000821	0.001841

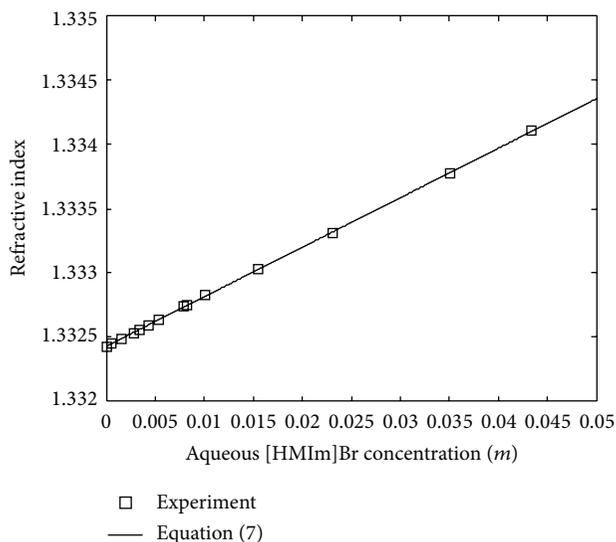


FIGURE 5: Variation in the refractive index of aqueous solutions of [HMIm]Br with concentration at 298.15 K and 0.1 MPa.

molar volume generally indicates contraction in volumes upon mixing. It can be due to the presence of hydrogen bonding. Figures 8 and 9 show the plot of excess molar volumes and excess refractive index versus concentrations, respectively. The values of excess volume and excess refractive index were reported in Table 4.

4. Conclusion

In the present work density, refractive index, and water activity of [HMIm]Br in aqueous solutions at 298.15 K and 0.1 MPa were determined experimentally at Reprocessing R&D Division. Based on the experimental results, apparent molal volume, vapour pressure, and excess properties have been estimated and reported. At infinity dilution there

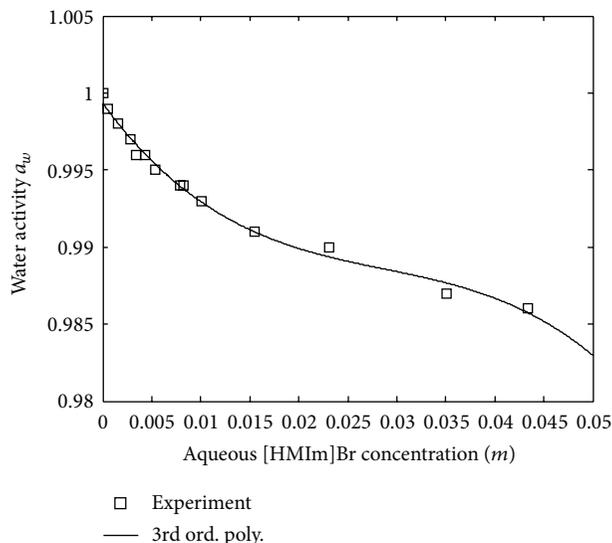


FIGURE 6: Variation of water activity with the concentration in the aqueous solutions of [HMIm]Br at 298.15 K at 0.1 MPa.

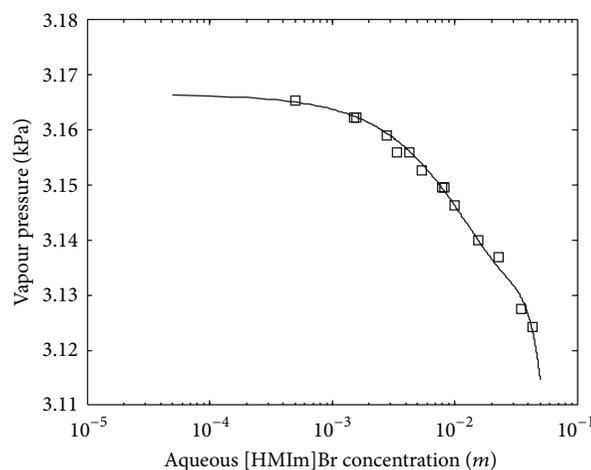


FIGURE 7: Vapour pressure of aqueous solutions of [HMIm]Br at 298.15 K.

will be no solute-solute interaction. Therefore $V_{2,\phi}^0$ gives information about solute-solvent interaction while a_v will provide information about solute-solute interaction. In the present study, $V_{2,\phi}^0$ and a_v both are positive, which indicates that both ion-solvent and ion-ion interactions are there, but as we have taken dilute solution, $V_{2,\phi}^0$ value is higher than a_v value indicating strong ion-solvent interaction. As ion-solvent interaction is high in comparison to ion-ion interaction, the salts will act as structure breakers in that concentration range. The negative values of excess molal volume show the significant deviations from ideality. The negative excess molar volumes of ([HMIm]Br + water) reflect the compactness of solution, that is, existence of net attractive interaction between the dissimilar molecules which could be due to the hydrogen bonding interactions.

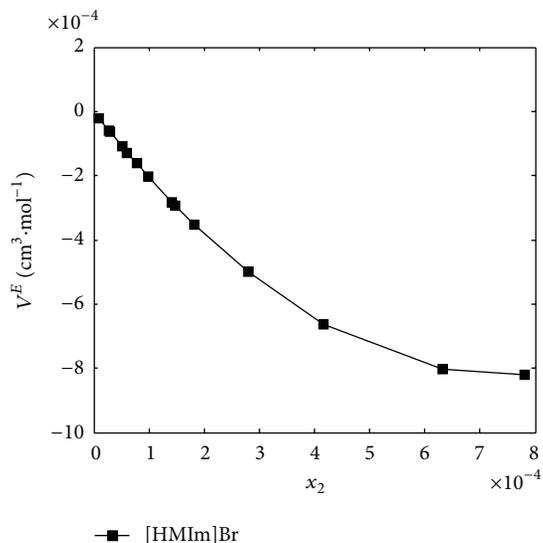


FIGURE 8: Plot of excess molar volume versus mole fraction concentration of aqueous solution of [HMIm]Br solutions at 298.15 K and 0.1 MPa.

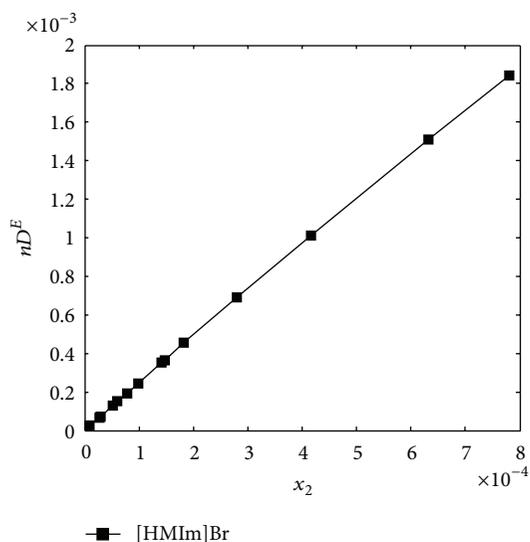


FIGURE 9: Plot of excess refractive index versus mole fraction concentration of aqueous solution of [HMIm]Br solutions at 298.15 K and 0.1 MPa.

Nomenclature

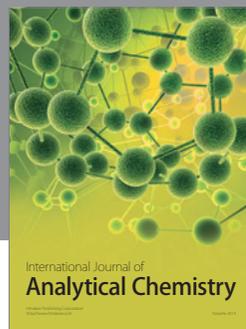
ρ^0 :	Density of water
ρ :	Density of aqueous solution
ρ_1 :	Density of pure [HMIm]Br
m :	Molality
V :	Total volume of the solution
n_1 :	Number of moles of water
n_2 :	Number of moles of the solute
V_1^0 :	Partial molal volume of pure component
$V_{2,\phi}$:	Apparent molal volume

$V_{2,\phi}^0$:	The limiting apparent molal volume
a_v :	Empirical constant
nD :	Predicted refractive index of solution
nD_w :	Refractive index of water at sodium d-line
a :	Coefficient of refractive index correlation
b :	Coefficient of refractive index correlation
α :	Cubic expansion coefficient (thermal isobaric expansibility)
V_m^E :	Excess molar volume
V_m :	Molar volume of solution
V_m^{ideal} :	Molar volume of pure components
M_1 :	Molar mass of water
M_2 :	Molar masses of [HMIm]Br
x_1 :	Mole fraction of water
x_2 :	Mole fraction of [HMIm]Br
A_i :	Adjusted coefficients for excess molar volume
Subscript 2:	Solute
Subscript 1:	Solvent.

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