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Densities, Excess Molar Volumes, and Refractive Properties of the Binary Mixtures of the Amino Acid Ionic Liquid [bmim][Gly] with 1-Butanol or Isopropanol at T = (298.15 to 313.15) K

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ABSTRACT: Densities and refractive indices were determined for two ionic liquid (IL) mixtures formed by 1-butyl-3-methylimidazolium glycine acid salt ([bmim][Gly]), with 1-butanol or isopropanol, respectively, over the mole fraction range from (0.1 to 0.9) and at temperatures from (298.15 to 313.15) K at intervals of 5 K and atmospheric pressure. Excess molar volumes and refractive index deviations have been calculated from the experimental data and fitted to a Redlich−Kister polynomial function. The excess volumes for the two systems are negative over both the composition and temperature ranges, whereas the refractive index deviations are always positive. Finally, results have been interpreted in terms of molecular interactions and molecular structures in these binary mixtures.

1. INTRODUCTION

Mixing different organic liquids gives rise to solutions that generally do not behave ideally. The thermodynamic and transport properties of liquids and liquid mixtures[1] are used to study the molecular interactions between the various components of the mixture and also to understand engineering applications concerning heat transfer, mass transfer, and fluid flow. Properties such as the density, viscosity, and refractive index of binary liquid mixtures over the whole concentration range are useful for a full understanding of their thermodynamic and transport properties and important to achieve for a theoretical and practical understanding.

A number of researchers have studied some of properties of ionic liquids (ILs) such as density or viscosity. The presence of water or alcohol in ILs has a large effect on physical properties.[2−4] There are a number of papers concerning the thermodynamics properties of binary systems of ILs.[5−7] A few successful samples have been reported; amino acid ILs are candidates to act as a platform for functional pure amino acid ILs in large quantities at low cost. Despite their importance and interest, details of the physicochemical properties of amino acid ILs have not been studied systematically. Particularly, properties of the mixtures of ILs with organic molecular liquids that are paramount for the design of many technological processes are very limited. In a previous article,[8] we reported experimental thermodynamic properties for binary 1-butyl-3-methylimidazolium glutamic acid salt ([bmim][Glu]) or 1-butyl-3-methylimidazolium glycine acid salt ([bmim][Gly]), respectively, with benzylalcohol. The present article follows such work, reporting values of densities and refractive indices, of binary mixtures of [bmim][Gly] with 1-butanol or isopropanol, respectively, at (298.15 to 313.15) K and at atmospheric pressure. From these experimental results, excess molar volume and refractive index deviations from the ideal behavior were calculated.

2. EXPERIMENTAL SECTION

Chemicals. The ILs ([bmim][Gly]) were prepared and purified by using the procedures described by Fukumoto et al.[9] (see Figure 1).

In the process above, the ILs were washed several times with dichloromethane and anhydrous ether to decrease the bromine content. It was determined that no precipitation of AgBr occurred by the addition of $AgNO_3$ to the wash liquid. To reduce the water content and volatile compounds (methylimidazole, dichloromethane, and anhydrous ether) to negligible values, vacuum (< 1.0 kPa) and moderate temperature to 353 K were applied to the [bmim][Gly] samples for several days, always immediately prior to their use. The water content of the [bmim][Gly], analyzed by Karl Fisher titration(ZSD-2 type), mass fraction of water was $1.3 \cdot 10^{-3}$. The ILs were dehydrated under high vacuum, and the structure of IL was identified by ^{1}H NMR (AVANCE III 400 MHz digital NMR spectrometer) and in good agreement with the literature;[10] a detailed formula is shown in Figure 2. From the NMR, the total peak integral in the ^{1}H NMR spectrum was found to correspond for all ILs to a nominal purity higher than 99 %. The bromine contents were measured by titration with $AgNO_3$, and the obtained values were below the detection limit (< 200 ppm).

1-Butanol and isopropanol (analytical reagent grade, mass fraction > 0.99), used in the experiments, were purchased from Shanghai Chemical Factory. They were further purified by fractional distillation until the boiling point and density were constant for successive fractions. Purities of chemicals used in this work are also given in Table 1. The purities of the liquid samples were tested using a gas chromatograph. The verified purities are in both cases > 99.5 %. To check the purity of the substances, the density and refractive index of *n*-butyl alcohol and isopropanol were determined at T = (298.15 to 313.15) K and compared with the literature values,[11−15] as listed in Table 1. It can be observed that the agreement is good.

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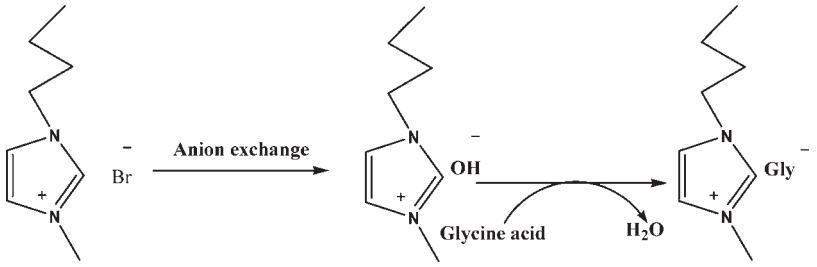

Figure 1. Preparation of the amino acid IL [bmim][Gly] by the neutralization method: 1, [bmim][Br]; 2, [bmim][OH]; 3, ([bmim][Gly].

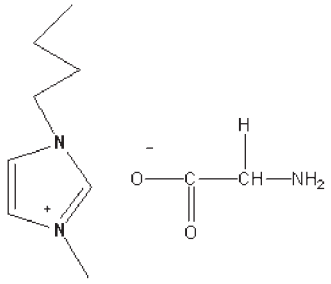

Figure 2. Molecular structure of the IL 1-butyl-3-methylimidazolium glycine acid salt ([bmim][Gly]).

Table 1. Densities ρ, Refractive Indices n_D of the Pure Components, and Comparison with the Literature Values

component	purity (%)	T/K	ρ/g·cm^{-3} exptl	ρ/g·cm^{-3} lit.	n_D exptl	n_D lit.
1-butanol	99.5	298.15	0.8060	0.8059[11]	1.3969	1.3967[13]
		303.15	0.8024	0.8024[12]	1.3951	
		308.15	0.7982	0.7981[11]	1.3928	1.3926[13]
		313.15	0.7946	0.7947[12]	1.3905	
isopropanol	99.5	298.15	0.7812	0.7811[14]	1.3751	1.3752[15]
		303.15	0.7740	0.7740[14]	1.3738	
		308.15	0.7658		1.3727	
		313.15	0.7572		1.3715	
[bmim][Gly]	99	298.15	1.26026		1.5202	
		303.15	1.25720		1.5188	
		308.15	1.25410		1.5171	
		313.15	1.25077		1.5166	

Apparatus and Procedure. Binary mixtures were prepared by mass in airtight glass bottles. The mass measurements were performed on a precise analytical balance (model: FA2014, SPSIC, China) with an accuracy of $1 \cdot 10^{-5}$ g. The required properties were measured on the same day of the mixture. Caution was taken to prevent evaporation, and the overall experimental uncertainty in the mole fraction composition is estimated to be less than $\pm$ $1 \cdot 10^{-4}$. Conversion to molar quantities was based on the relative atomic mass table of 2006 issued by the International Union of Pure and Applied Chemistry (IUPAC).[16]

Densities, ρ, of the pure compounds and their mixtures were determined with an Anton Paar DMA4500 vibrating

Table 2. Densities, Refractive Indices, Excess Molar Volumes, and Refractive Index Deviations for Binary Mixtures at the Temperatures from (298.15 to 313.15) K and Atmospheric Pressure

x_1	ρ / g·cm^{-3}	n_D	V_m^E / cm^3·mol^{-1}	100 Δn_D
	x([bmim][Gly]) + (1 − x) 1-Butanol			
		T = 298.15 K		
0.0972	0.8852	1.4176	−0.460	0.869
0.2125	0.9625	1.4384	−0.651	1.529
0.3041	1.0141	1.4536	−0.637	1.919
0.3849	1.0543	1.4649	−0.593	2.054
0.4929	1.1014	1.4775	−0.481	1.986
0.6103	1.1463	1.4900	−0.367	1.788
0.6923	1.1742	1.4970	−0.287	1.475
0.8037	1.2084	1.5061	−0.174	1.013
0.9129	1.2384	1.5143	−0.068	0.484
		T = 303.15 K		
0.0972	0.8817	1.4169	−0.482	0.975
0.2125	0.9591	1.4380	−0.682	1.659
0.3041	1.0109	1.4522	−0.692	1.944
0.3849	1.0512	1.4632	−0.647	2.051
0.4929	1.0984	1.4762	−0.536	2.017
0.6103	1.1433	1.4883	−0.414	1.769
0.6923	1.1712	1.4960	−0.323	1.522
0.8037	1.2054	1.5051	−0.201	1.061
0.9129	1.2354	1.5126	−0.082	0.459
		T = 308.15 K		
0.0972	0.878	1.4154	−0.503	1.050
0.2125	0.955	1.4365	−0.714	1.729
0.3041	1.007	1.4504	−0.734	1.979
0.3849	1.048	1.4607	−0.683	2.008
0.4929	1.095	1.4743	−0.581	2.027
0.6103	1.140	1.4869	−0.456	1.822
0.6923	1.168	1.4947	−0.366	1.580
0.8037	1.202	1.5040	−0.226	1.128
0.9129	1.232	1.5116	−0.084	0.529
		T = 313.15 K		
0.0972	0.874	1.4130	−0.517	1.029
0.2125	0.952	1.4345	−0.756	1.720
0.3041	1.004	1.4595	−0.773	2.062
0.3849	1.044	1.4608	−0.731	2.179

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Table 2. Continued

x_1	ρ / g·cm^{-3}	n_D	V_m^E / cm^3·mol^{-1}	100 Δn_D
0.4929	1.092	1.4735	−0.640	2.085
0.6103	1.137	1.4858	−0.507	1.837
0.6923	1.165	1.4936	−0.416	1.584
0.8037	1.199	1.5027	−0.267	1.084
0.9129	1.229	1.5104	−0.114	0.482
x_1([bmim][Gly]) + (1 − x_1) Isopropanol				
T = 298.15 K				
0.0965	0.8776	1.4050	−0.514	1.589
0.1992	0.9578	1.4303	−0.713	2.634
0.3059	1.0244	1.4508	−0.757	3.130
0.3977	1.0719	1.4657	−0.743	3.289
0.5115	1.1216	1.4796	−0.703	3.027
0.5868	1.1499	1.4876	−0.666	2.733
0.7052	1.1884	1.4989	−0.550	2.145
0.8117	1.2175	1.5085	−0.366	1.558
0.8775	1.2335	1.5143	−0.230	1.192
T = 303.15 K				
0.0965	0.8707	1.4036	−0.521	1.580
0.1992	0.9514	1.4294	−0.729	2.674
0.3059	1.0186	1.4498	−0.780	3.165
0.3977	1.0665	1.4640	−0.766	3.249
0.5115	1.1168	1.4781	−0.738	3.017
0.5868	1.1454	1.4868	−0.691	2.794
0.7052	1.1843	1.4989	−0.565	2.287
0.8117	1.2139	1.5075	−0.386	1.604
0.8775	1.2300	1.5126	−0.235	1.160
T = 308.15 K				
0.0965	0.8630	1.4029	−0.544	1.625
0.1992	0.9442	1.4287	−0.758	2.725
0.3059	1.0120	1.4489	−0.805	3.208
0.3977	1.0606	1.4630	−0.798	3.288
0.5115	1.1114	1.4773	−0.755	3.078
0.5868	1.1405	1.4855	−0.714	2.809
0.7052	1.1800	1.4972	−0.592	2.267
0.8117	1.2100	1.5049	−0.392	1.501
0.8775	1.22640	1.5099	−0.244	1.048
T = 313.15 K				
0.0965	0.8547	1.4024	−0.555	1.685
0.1992	0.9366	1.4272	−0.778	2.678
0.3059	1.0050	1.4488	−0.825	3.288
0.3977	1.0541	1.4624	−0.813	3.315
0.5115	1.1058	1.4772	−0.783	3.147
0.5868	1.1353	1.4852	−0.741	2.855
0.7052	1.1754	1.4965	−0.618	2.269
0.8117	1.2059	1.5056	−0.414	1.636
0.8775	1.2226	1.5104	−0.258	1.162

tube densimeter automatically thermostatted within ± 0.01 K. The densimeter was calibrated with deionized double-distilled water, heptane, octane, isooctane, cyclohexane, and benzene, using ρ values from the literature.[15,17] After each series of measurements, the densimeter should be washed by two kinds of lotions and dried. The experimental technique was checked by determining V_m^E of the standard mixtures: (cyclohexane + benzene) at the temperatures 293.15 K. Our results agree well with published values.[18] The uncertainty of our density data was estimated to be ± 5·10^{-4} g·cm^{-3}. The accuracy in V_m^E is believed to be less than ± (0.01|V_{max}^E| + 0.005) cm^3·mol^{-1}, where |V_{max}^E| denotes the maximum experimental value of the excess molar volume with respect to the mole fraction.

Refractive indices of the mixtures at the sodium D-line were determined with an Abbe refractometer equipped with a circulating water bath permitting the sample to be maintained at a constant temperature to within ± 0.01 K. The instrument was calibrated by measuring the refractive index of deionized water. The sample support was rinsed with acetone and dried with a paper towel. The uncertainty in refractive index measurements, without the uncertainty of temperature, was found to be 5·10^{-4}.

At least three repetitions of each data set were obtained, and the results were averaged.

Table 3. Parameters and Standard Deviations, σ, of Equations 3 and 4 for Binary Mixtures at Different Temperatures

	a_0	a_1	a_2	a_3	σ
x_1([bmim][Gly]) + (1 − x_1) 1-Butanol					
T = 298.15 K					
V_m^E/cm^3·mol^{-1}	−1.911	−2.017	−1.782	−1.071	0.005
100 Δn_D	7.981	2.610	−0.055	−0.444	0.029
T = 303.15 K					
V_m^E/cm^3·mol^{-1}	−2.122	−2.089	−1.761	−1.015	0.004
100 Δn_D	8.040	2.368	0.743	1.337	0.0157
T = 308.15 K					
V_m^E/cm^3·mol^{-1}	−2.309	−2.018	−1.706	−1.319	0.004
100 Δn_D	8.033	2.084	2.048	1.841	0.021
T = 313.15 K					
V_m^E/cm^3·mol^{-1}	−2.521	−2.048	−1.788	−1.084	0.003
100 Δn_D	8.389	2.723	0.826	1.083	0.025
x_1([bmim][Gly]) + (1 − x_1) Isopropanol					
T = 298.15 K					
V_m^E/cm^3·mol^{-1}	−2.844	−0.794	−1.694	−2.490	0.003
100 Δn_D	12.134	6.233	3.842	−2.946	0.041
T = 303.15 K					
V_m^E/cm^3·mol^{-1}	−2.959	−0.794	−1.627	−2.504	0.004
100 Δn_D	12.358	5.505	3.396	−1.504	0.033
T = 308.15 K					
V_m^E/cm^3·mol^{-1}	−3.057	−0.813	−1.719	−2.680	0.003
100 Δn_D	12.541	5.712	2.528	−0.119	0.036
T = 313.15 K					
V_m^E/cm^3·mol^{-1}	−3.155	−0.758	−1.779	−2.768	0.003
100 Δn_D	12.612	5.453	3.665	−0.400	0.037

3. RESULTS AND DISCUSSION

The experimental values of the density and refractive index for binary mixtures at different temperatures and at atmospheric

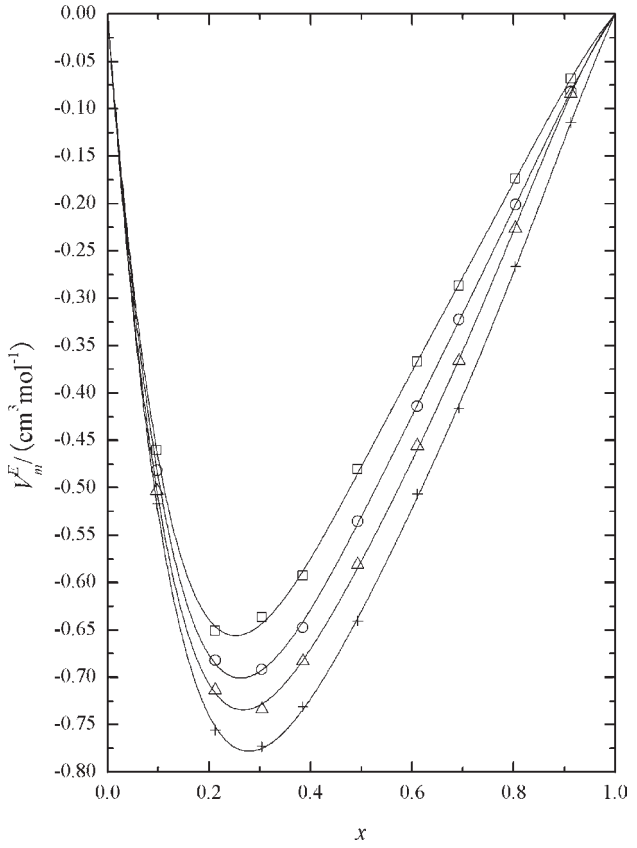

Figure 3. Excess molar volume vs mole fraction for $\{x_1$[bmim][Glu] + $(1 - x_1)$ 1-butanol$\}$ mixtures: □, T = 298.15 K; ○, T = 303.15 K; △, T = 308.15 K; +, T = 313.15 K.

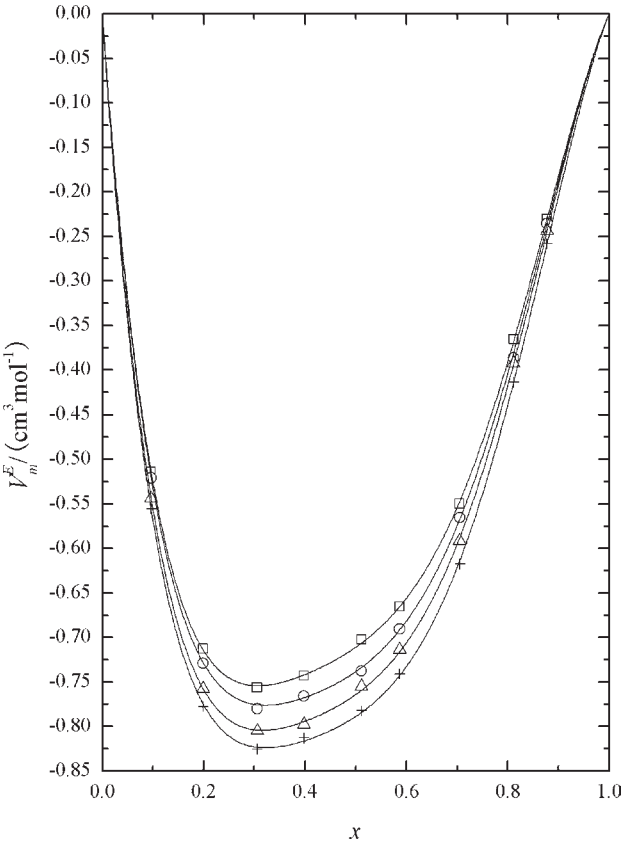

Figure 4. Excess molar volume vs mole fraction for $\{x_1$[bmim][Gly] + $(1 - x_1)$ isopropanol$\}$ mixtures: □, T = 298.15 K; ○, T = 303.15 K; △, T = 308.15 K; +, T = 313.15 K.

pressure are listed in Table 2. Excess molar volumes were calculated from our experimental data according to the following equation:

$$V_m^E = x_1 M_1(1/\rho - 1/\rho_1) + x_2 M_2(1/\rho - 1/\rho_2) \quad (1)$$

where x_1 and x_2 are the mole fractions of components 1 and 2, ρ_1 and ρ_2 are the densities of pure components 1 and 2, ρ is the density of the mixture, and M_1 and M_2 are the molecular weights of components 1 and 2.

The refractive index deviations, Δn_D, at various temperatures from the linear additive values of the mole fractions were calculated by means of the eq 3:

$$\Delta n_D = n_D - (x_1 n_{D1} + x_2 n_{D2}) \quad (2)$$

where n_D is the refractive index of the mixture and n_{D1} and n_{D2} are the refractive indices of components 1 and 2, respectively.

The values of V_m^E and Δn_D of the studied binary mixtures, at all measured temperatures, are given in Table 2. The significant digits were determined taking into account each experimental error.

The excess molar volume and refractive index deviations were fitted by means of a Redlich−Kister type equation[19] of the form:

$$Y^E = x_1(1 - x_1)\sum_{j=0}^{j=n} a_j(1 - 2x_1)^j \quad (3)$$

where Y^E represent either V_m^E or Δn_D, a_j are adjustable parameters, and x_1 is the mole fraction of ([bmim][Gly]). The number of coefficients used for each property was determined as the minimum number needed to adequately represent the data. The standard deviation, σ, between the experimental and calculated values was defined as:

$$\sigma = [\sum(Y_{exptl}^E - Y_{calcd}^E)^2/(N - p)]^{1/2} \quad (4)$$

where N and p are the number of direct experimental values and parameters, respectively.

The values of the parameters a_j together with the standard deviation σ for each property Y^E are given in Table 3. Excess molar volume, V_m^E, and refractive index deviations, Δn_D, are graphically represented in Figures 3 to 6, respectively. No data have been encountered in the literature for comparison.

As can be seen in Figures 3 and 4, the V_m^E values for binary mixtures of [bmim][Gly] with 1-butanol or isopropanol are negative over the whole composition range, following the sequence: 1-butanol > isopropanol, and decrease slightly with temperature. The figures are asymmetrical, and in the mole fraction of 0.3 there occurs a minimum for both of the two systems.

Excess molar volumes, which are a measure of the deviations of the actual property from the property if the system behaves ideally, give information on molecular interactions between the component molecules of the mixture and are influenced by effects such as differences in shape and size of the component molecules, reorientation of the component molecules in the mixture, and intermolecular interactions.[20,21] It is well-known

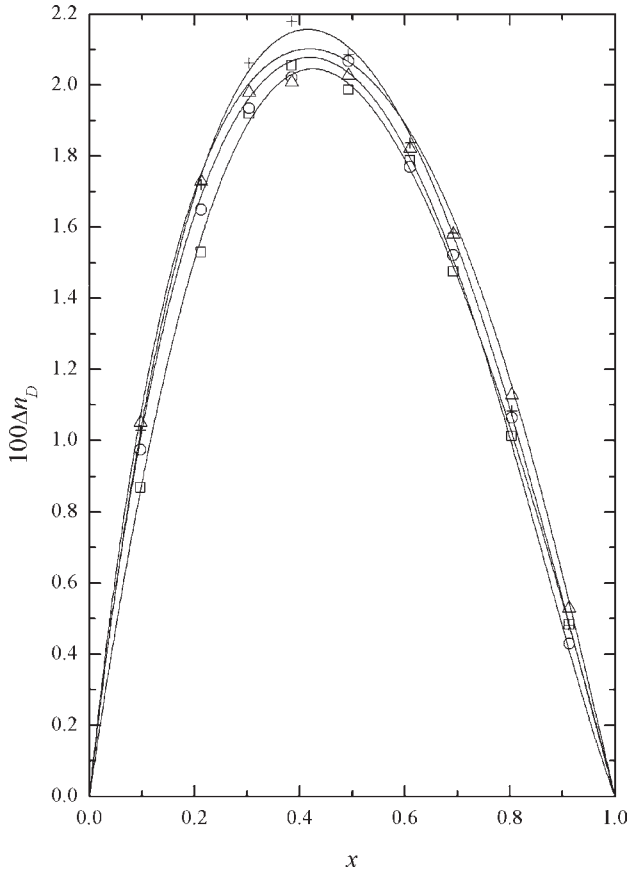

Figure 5. Refractive index deviations Δn_D vs mole fraction for $\{x_1$[bmim][Gly] + $(1 - x_1)$ 1-butanol$\}$ mixtures: □, T = 298.15 K; ○, T = 303.15 K; △, T = 308.15 K; +, T = 313.15 K.

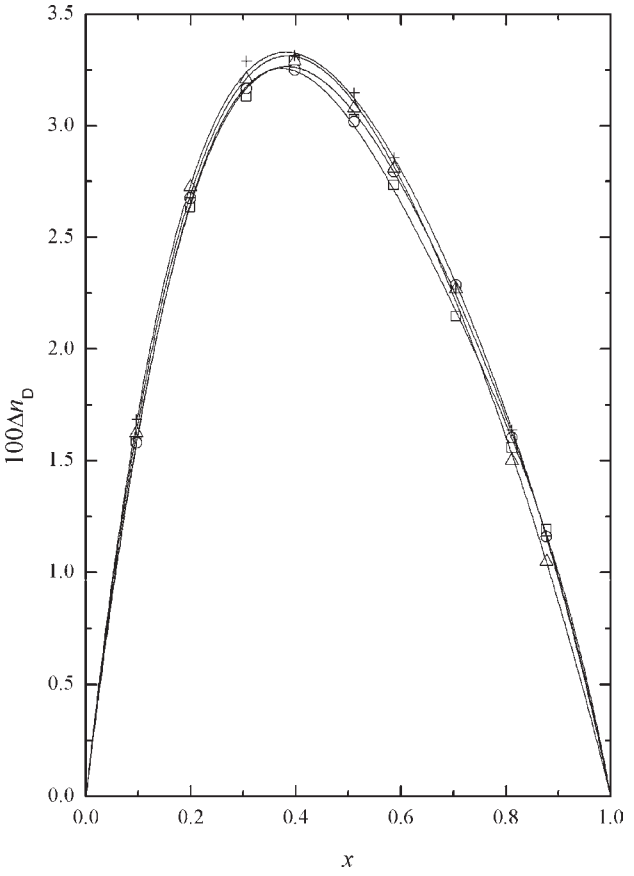

Figure 6. Refractive index deviations Δn_D vs mole fraction for $\{x_1$[bmim][Gly] + $(1 - x_1)$ isopropanol$\}$ mixtures: □, T = 298.15 K; ○, T = 303.15 K; △, T = 308.15 K; +, T = 313.15 K.

that V_m^E is the result of several opposing effects. Interactions between like molecules lead to increased V_m^E values, while negative contributions to V_m^E arise from interactions between unlike molecules, or structural effects as changes in free volume, or interstitial accommodation. For the investigated systems, the negative V_m^E values determined here may be due to interactions between unlike molecules. Fukumoto et al.[9] have reported that the hydrogen bonding interaction between alkanol and amino acid IL is the main interaction on this kind of systems. Our results support the conclusion.

Comparing V_m^E values of 1-butanol and isopropanol, the V_m^E of the mixture containing isopropanol is smaller than that of containing 1-butanol. This may be interpreted assuming a more efficient packing occurred for isopropanol than 1-butanol. A similar phenomenon has been observed for ([bmim][BF_4] + benzaldehyde) by Qi et al.[22]

The deviation of refractive indices Δn_D are positive over the whole composition range, with the maxima lying nearly at $x_1 \approx 0.4$. Figures 5 and 6 show the deviation of refractive indices Δn_D at various temperatures of the mixture of [bmim][Gly] with 1-butanol or isopropanol; the values increase as the temperature increases. Contrary to V_m^E, the Δn_D of the mixture containing 1-butanol is smaller than that of containing isopropanol.

4. CONCLUSION

In the present work, the densities and refractive indices of two binary systems, ([bmim][Gly] + 1-butanol) and ([bmim][Gly]+ isopropanol), have been measured experimentally over temperatures from (298.15 to 313.15) K and at atmospheric pressure. The experimental density values were then used for the calculation of excess molar volumes, which were then correlated using a Redlich−Kister-type polynomial equation. The densities of both binary systems decreased with increasing temperature, whereas the excess molar volumes were totally negative. The calculated refractive index deviations, Δn_D, for the two mixtures are all positive at all temperatures and increase when temperature increases from (298.15 to 313.15) K.

The present results add useful data on amino acid IL to the growing database on IL properties, a database that is essential for the many applications of these liquids currently under exploration.

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