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Densities and Viscosities of 1-Butyl-3-methylimidazolium Tetrafluoroborate + H_2O Binary Mixtures from (303.15 to 353.15) K

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Densities and viscosities of water (1) + 1-butyl-3-methylimidazolium tetrafluoroborate (2) were measured over the entire mole fraction range from (303.15 to 353.15) K. From these data, excess molar volumes (V^E), and viscosity deviations ($\Delta\eta$) were calculated. The V^E and $\Delta\eta$ were fitted to the Redlich−Kister equation using a multiparametric nonlinear regression method. Estimated coefficients of the Redlich−Kister equation and standard deviation calculated from the Redlich−Kister equation to the experimental data are also presented. The results show that the densities and viscosities are dependent strongly on water content. Comparatively, the viscosity deviation $\Delta\eta$ is more sensitive to temperature than the excess molar volume V^E.

Introduction

Ionic liquids (ILs) are low melting point salts that represent an exiting new class of reaction solvents. Being composed entirely of ions, they negligible vapor pressure and can be prepared from a wide range of cations and anions. The are also miscible in water, alcohol, etc. depending on anions and cations. The key attraction of ILs is their possibility to be tuned by varying the nature of the cations and anions. Because of this combination of properties, ILs present many possible applications in many fields, such as organic synthesis, catalytic reactions, and multiphase separations. An ionic liquid that did not hydrolyze was reported by Wilkes and Zaworotko in 1992.[1] Up to now, most work has investigated the synthetic methods and applications of ILs. Physicochemical and thermophysical properties have not been studied systematically. Densities and viscosities are needed for the design of new processes. The experimental data of density and viscosity of a binary mixture are important from the theoretical viewpoint to understand the liquid theory. So a study on excess thermophysical properties is important in order to predict the properties and characteristics of ILs.[2]

Several properties of some 1-alkyl-3-methylimidazolium tetrafluoroborates have been reported in the literature,[3−6] including 1-butyl-3-methylimidazolium tetrafluoroborate ([BMIM][BF_4]). However, the later is less reported than other imidazolium-based ILs. Most researchers reported the data of its density, viscosity, and conductivity at room temperature.[7−12] Its densities over a temperature range from (295 to 343) K were reported by Christopher et al.[8] Kim et al.[9,10] have given its heat capacities over a temperature range from (298 to 323) K. Moreover, the vapor pressure of a [BMIM][BF_4] + water mixture was also reported.[11] The author[4] just showed figures about density at 40 °C and viscosity at 20 °C of mixtures [BMIM][BF_4] and water. Zhang et al.[13] has reported density and viscosity data of mixtures of [EMIM][BF_4] and water from (293.15 to 323.15) K. In these literatures, density and viscosity data of the mixtures of [BMIM][BF_4] and water are never mentioned.

In this work, the densities and viscosities of mixtures consisting of 1-butyl-3-methylimidazolium tetrafluoroborate and water were measured as a function of compositions from (303.15 to 353.15) K. The excess molar volume V^E and the viscosity derivation $\Delta\eta$ of this binary system have been obtained and fitted to the Redlich−Kister equation. The effects of water content and temperature on the physical properties were analyzed, and the interactions and structures were discussed in terms of the behavior of excess molar volume V^E of the mixture of the [BMIM][BF_4] and water.

Experimental Section

Materials. In this work, all the aqueous solutions were prepared with deionized water. The IL (1-butyl-3-methylimidazolium tetrafluoroborate) was prepared according to following method.

Preparation of [BMIM][BF_4]. The chemicals with preparation of IL (1-butyl-3-methylimidazolium tetrafluoroborate) were of analytical grade and used without further purification.

First, 1-butyl-3-methylimidazolium bromide was synthesized. Butyl bromide (1.1 mol) was added dropwise into 1-methylimidazole (1 mol) with agitation at 343 K. The reaction mixture was refluxed for 24 h, and when cooled to room temperature, ethyl acetate was added to the mixture. The ethyl acetate was removed by a separating funnel followed by the addition of fresh ethyl acetate, and this step was repeated four times. The remaining ethyl acetate was removed by rotary evaporation, and the solution was dried under high vacuum at (343 to 353) K for at least 6 h to get 1-butyl-3-methylimidazolium bromide ([BMIM][Br]) at very high yield (99 %).

Second, the tetrafluoroborate salt was prepared by metathesis reactions from the corresponding bromide. [BMIM][Br] (0.1 mol) was dissolved in acetonitrile (50 mL), and ammonium tertrafluorobarate (0.11 mol) was added. The mixture was refluxed for at least 24 h. When it was cooled to room temperature, NH_4Br precipitate was removed by filtration. Any remaining precipitate was removed by further filtration at this step. The remaining acetonitrile was removed by rotary evaporation to get crude 1-butyl-3-methylimidazolium tetrafluorobarate. Crude [BMIM][BF_4] was dissolved in dichloromethane (50 mL) and cooled below 5 K. Deionized water and a separation funnel were also cooled to below 5 K. The dichloromethane solution was washed with cooled deionized water

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Table 1. Comparison of Measured Densities ρ and Viscosities η of the [BMIM][BF$_4$] with Literature Values

	ρ/g·cm^{-3}		η/mPa·s	
T/K	exp	lit	exp	lit
293.15	1.2046^a		122.35^a	154[12] 233[7]
298.15	1.2015^a	1.20[11]	94.26^a	180[6] 279.86[10]
303.15	1.1984	1.17[7] 1.2005[8] 1.20[11]	74.21	
308.15	1.1954	1.19[11]	58.18	
313.15	1.1922	1.1940[8] 1.19[11]	46.51	
318.15	1.1890	1.19[11]	37.79	
323.15	1.1860	1.18[11]	31.08	
333.15	1.1798		21.52	
343.15	1.1735	1.1737[8]	15.61	
353.15	1.1647		11.49	

[a] The value of density and viscosity of [BMIM][BF$_4$] was extrapolated.

(30 mL) five times until the aqueous solution did not form any precipitate with 0.1 mol·L^{-1} AgNO$_3$ solution. The solvent dichloromethane was removed by rotary evaporation, and the [BMIM][BF$_4$] was dried under high vacuum at (323 to 333) K for at least 6 h.

The chemical shift for ^{1}H NMR spectrum (parts per million, D$_2$O) appear as follows: δ 8.575 [s, 1H, H(2)], 7.390 [s, 1H, H(4)], 7.350 [s, 1H, H(5)], 4.133 [t, 2H, NCH$_2$], 3.817[s, 3H, NCH$_3$], 1.809 [m, 2H, NCH$_2$-CH$_2$], 1.295 [m, 2H, NCH$_2$CH$_2$-CH$_2$], and 0.853 [t, 3H, CH$_3$]. The ^{13}C NMR spectrum (parts per million, D$_2$O) contains peaks: 138.625 [C(2)], 126.155 [C(4)], 124.861 [C(5)], 51.931 [N-CH$_2$], 38.233 [NCH$_3$], 33.980 [NCH$_2$-CH$_2$], 21.445 [NCH$_2$CH$_2$-CH$_2$], and 15.288 [CH$_3$]. Impurity peaks were not observed in the ^{1}H NMR and ^{13}C NMR spectrums, and there is a solvent peak at $\delta = 4.653$ in ^{1}H NMR spectrum. The chemical shift of other peaks corresponded to structure of the [BMIM][BF$_4$]. Water content of the product [BMIM][BF$_4$] was determined to be about 200 ppm by the Karl Fischer titration. The purity of the ionic liquid was > 99 %. The difference of pH values of this IL between room temperature and 353 K was less than 1 %. This phenomenon represented that this ionic liquid will not hydrolyze at 353 K. Its densities at several temperatures are in a good agreement with those reported in the literature (in the Table 1).

Apparatus and Procedure. The densities of the IL [BMIM][BF$_4$] and its mixtures were measured with a pycnometer (10 mL), and its capacities varied with the experiment temperature and were calibrated with pure water. The temperature was controlled by a constant water bath with an accuracy of ± 0.05 K. The uncertainty of the corresponding density measurement was within ± 0.0001.

The mixtures of [BMIM][BF$_4$] and water were prepared by mass using a JA2003 electronic digital balance accurate to within ± 0.1 mg. The uncertainty in the mole fraction of the mixtures was estimated to less than ± 0.0001. The uncertainty of excess molar volumes was estimated better than ± 0.02. All molar quantities are based on the IUPAC relative atomic mass table.

Measurements of viscosity and density were carried out in the same water bath. The viscosities of mixture were measured using an Ubbelohde capillary viscometer. The capillary was calibrated for kinetic energy correction with double-distilled water and 1,4-butanediol at the experimental temperature range:

$$\nu = \frac{\eta}{\rho} = k_1 t - k_2/t \tag{1}$$

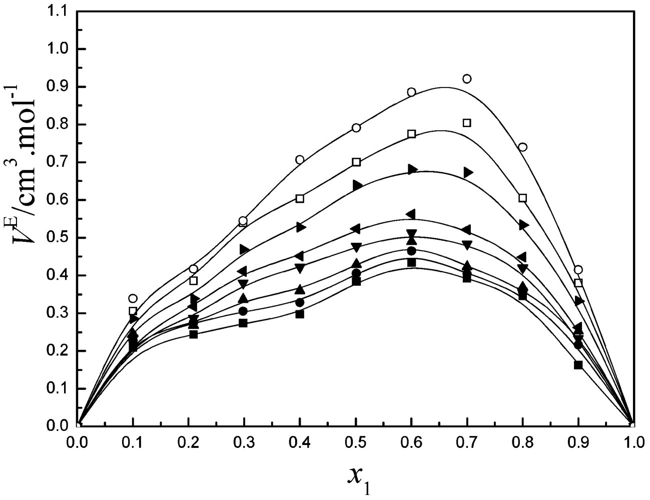

Figure 1. Excess molar volume V^E vs x mole fraction of water for water (1) + [BMIM][BF$_4$] (2). ■, 303.15K; ●, 308.15K; ▲, 313.15K; ▼, 318.15K; left-facing solid triangle, 323.15K; right-facing solid triangle, 333.15K; □, 343.15K; ○, 353.15K. The symbols represent experimental values.

where t is the flow time. The kinematic viscosity ν for calibration was obtained from literature values of the absolute viscosity and density.[14,15] Two viscometers were used for all measurements. One of the capillaries was (0.6 to 0.7) mm in diameter and 120 mm in length for mole fraction of water from (0 to 0.5); the other one was (0.1 to 0.2) mm in diameter and 100 mm in length for mole fraction from (0.6 to 1.0). Therefore, the end correction could be neglected. An electronic digital stopwatch with a readability of ± 0.01 s was used for flow time measurement. Experiments were repeated at least four times at each temperature for all mixtures, and the results were averaged.

The overall uncertainty of viscosity measurements depends on equilibrium temperature, flow time, mole fraction, and calibration fluid. So according to the precision of all experimental instruments, the experimental average uncertainties of viscosity were estimated to be ± 0.01. The uncertainties of viscosity derivations were about ± 0.02.

Result and Discussion

The densities and viscosities of mixtures, [BMIM][BF$_4$] and water, as a function of water content over the temperature range from (303.15 to 353.15) K are presented in Table 2. It can be readily observed that an increase in water content or temperature causes density or viscosity to decrease. The increase in temperature causes both the density and the viscosity to decrease slightly. Therefore, physical properties of ILs can be adjusted to meet the needs of applications for hydrophilic ionic liquids (e.g., [BMIM][BF$_4$]) by adding water or changing temperature.

The excess molar volumes V^E and the viscosity derivations $\Delta\eta$ were calculated from our measurements according to the following equations:[16−18]

$$V^E = \frac{x_1 M_1 + x_2 M_2}{\rho} - \frac{x_1 M_1}{\rho_1} - \frac{x_2 M_2}{\rho_2} \tag{2}$$

$$\Delta\eta = \eta - (x_1\eta_1 + x_2\eta_2) \tag{3}$$

where ρ and η are density and viscosity of mixtures; x_1 and x_2 are mole fractions; M_1 and M_2 are molar masses; ρ_1 and ρ_2 are densities; and η_1 and η_2 are the viscosities of water (1) and ionic liquid [BMIM][BF$_4$] (2), respectively.

Table 2. Experimental Densities ρ, Viscosities η, Excess Molar Volume V^E, and Viscosity Derivation $\Delta\eta$ for Binary Mixture of Water (1) + [BMIM][BF$_4$] (2)

	T/K							
x_1	303.15	308.15	313.15	318.15	323.15	333.15	343.15	353.15
				ρ/g·cm^{-3}				
0.0000	1.1984	1.1954	1.1922	1.1890	1.1860	1.1798	1.1735	1.1674
0.1002	1.1948	1.1917	1.1884	1.1853	1.1823	1.1758	1.1694	1.1631
0.2090	1.1915	1.1883	1.1852	1.1819	1.1787	1.1724	1.1658	1.1595
0.2984	1.1881	1.1849	1.1815	1.1780	1.1748	1.1682	1.1614	1.1553
0.4001	1.1833	1.1801	1.1767	1.1730	1.1698	1.1630	1.1561	1.1491
0.5014	1.1762	1.1731	1.1698	1.1662	1.1628	1.1555	1.1487	1.1417
0.6004	1.1670	1.1638	1.1605	1.1572	1.1537	1.1462	1.1389	1.1315
0.7000	1.1548	1.1519	1.1487	1.1448	1.1414	1.1331	1.1247	1.1163
0.8000	1.1347	1.1319	1.1289	1.1250	1.1217	1.1142	1.1068	1.0981
0.8999	1.0995	1.0955	1.0919	1.0899	1.0865	1.0789	1.0717	1.0647
1.0000	0.9957	0.9940	0.9922	0.9902	0.9881	0.9832	0.9778	0.9718
				η/mPa·s				
0.0000	74.21	58.18	46.51	37.79	31.08	21.52	15.61	11.49
0.1002	47.61	38.12	30.72	25.72	21.49	15.42	11.46	8.63
0.2090	34.89	28.35	23.50	19.64	16.74	12.02	8.98	6.79
0.2984	23.32	19.42	16.15	14.07	11.67	8.59	6.46	4.96
0.4001	16.57	13.57	11.44	9.72	8.41	6.16	4.68	3.56
0.5014	12.05	10.50	8.98	7.77	6.80	5.28	4.24	3.44
0.6004	10.05	8.69	7.40	6.40	5.69	4.75	3.75	3.19
0.7000	6.70	5.85	5.35	4.53	4.05	3.29	2.73	2.31
0.8000	4.36	3.80	3.36	2.99	2.68	2.19	1.82	1.54
0.8999	2.53	2.21	1.95	1.73	1.54	1.28	1.07	0.91
1.0000	0.80	0.72	0.65	0.60	0.55	0.47	0.40	0.35
				V^E/cm^3·mol^{-1}				
0.1002	0.21	0.23	0.25	0.23	0.24	0.29	0.31	0.34
0.2090	0.24	0.27	0.27	0.29	0.32	0.34	0.39	0.42
0.2984	0.27	0.30	0.34	0.38	0.41	0.47	0.54	0.54
0.4001	0.30	0.33	0.36	0.42	0.45	0.53	0.60	0.71
0.5014	0.38	0.41	0.43	0.48	0.52	0.64	0.70	0.79
0.6004	0.43	0.46	0.49	0.51	0.56	0.68	0.77	0.88
0.7000	0.39	0.40	0.42	0.48	0.52	0.67	0.82	0.98
0.8000	0.35	0.36	0.37	0.42	0.45	0.53	0.60	0.74
0.8999	0.16	0.22	0.25	0.23	0.26	0.33	0.38	0.41
				$\Delta\eta$/mPa·s				
0.1002	−19.3	−14.3	−11.2	−8.4	−6.5	−4.0	−2.6	−1.8
0.2090	−24.0	−17.8	−13.4	−10.4	−8.0	−5.1	−3.5	−2.4
0.2984	−29.0	−21.1	−16.1	−12.6	−10.3	−6.7	−4.6	−3.2
0.4001	−28.3	−21.6	−16.7	−13.2	−10.5	−6.9	−4.8	−3.5
0.5014	−25.4	−18.6	−14.5	−11.4	−9.0	−5.7	−3.8	−2.5
0.6004	−20.1	−15.0	−11.6	−9.1	−7.1	−4.1	−2.7	−1.6
0.7000	−16.1	−12.1	−9.1	−7.2	−5.7	−3.5	−2.2	−1.4
0.8000	−11.1	−8.4	−6.5	−5.1	−4.0	−2.5	−1.6	−1.0
0.8999	−5.6	−4.3	−3.3	−2.6	−2.1	−1.3	−0.9	−0.6

All values of V^E and $\Delta\eta$ for the mixtures of water (1) and [BMIM][BF$_4$] (2) were fitted to the Redlich−Kister polynominal equation:[19−21]

$$Y = x_1(1 - x_1)\sum_{i=0}^{m} A_i(2x_1 - 1)^i \tag{4}$$

where $Y = V^E$ or $\Delta\eta$, A_i are adjustable parameters, and x_1 is the mole fraction of water (1). The optimum number of coefficients A_i was determined from an examination of variation of standard derivation:

$$\sigma(Y) = [\sum(Y_{\mathrm{cal}} - Y_{\mathrm{exp}})^2/(n - m)]^1/^2 \tag{5}$$

n is the number of experimental data, and m is the number of coefficients of the Redlich−Kister equation.

The data of excess molar volume and viscosity derivation are shown in Table 2. The excess molar volume versus the mole fraction of water is plotted in Figure 1. Viscosity derivation versus mole fraction of water is shown in Figure 2. Table 3 lists the values of the parameters A_i together with the standard deviations.

The excess thermodynamic properties, which depend on the composition and/or temperature, are of great importance in understanding the nature of molecular aggregation that exists in the binary mixtures. The values of excess molar volume was positive for water + [BMIM][BF$_4$] mixtures at all temperatures and over the entire range of compositions. The excess molar volumes V^E increases slightly with increase in temperature from 303.15 K to 353.15 K. A maximum value was obtained at $x_1 = 0.7$.

The dependence of the viscosity derivations $\Delta\eta$ on mole fraction x_1 was displayed in Figure 2. The values of viscosity derivations were negative over the whole range of compositions, and the minimum existed at $x_1 = 0.3$ within the whole region of experimental temperatures. Simultaneously viscosity derivations increased slightly from (303.15 to353.15) K.

Conclusions

New experimental data of density and viscosity for the system of water + [BMIM][BF$_4$] were measured over the whole range of compositions from (303.15 to 353.15) K. The excess molar volume V^E and viscosity deviations $\Delta\eta$ of this system were correlated using the Redlich−Kister polynomial equation.

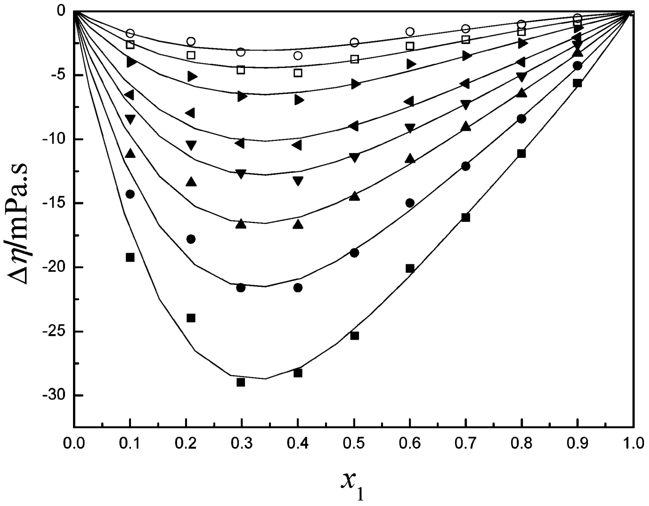

Figure 2. Viscosity derivation $\Delta\eta$ vs x mole fraction of water for water (1) + [BMIM][BF_4] (2). ■, 303.15K; ●, 308.15K; ▲, 313.15K; ▼, 318.15K; left-facing solid triangle, 323.15K; right-facing solid triangle, 333.15K; □, 343.15K; ○, 353.15K. The solid curves are calculated with the Redlich−Kister equation; the symbols represent experimental values.

Table 3. Coefficients of the Redlich−Kister Equation for V^E and $\Delta\eta$ of Water (1) + [BMIM][BF_4] (2) System

property	T/K	A_0	A_1	A_2	A_3	σ
V^E/cm^3·mol^{-1}	303.15	1.486	1.342	0.906	−2.571	0.012
	308.15	1.546	1.048	1.347	−1.764	0.017
	313.15	1.612	0.983	1.609	−1.453	0.025
	318.15	1.884	1.115	1.061	−1.678	0.014
	323.15	2.054	1.083	1.069	−1.379	0.012
	333.15	2.451	1.696	1.359	−2.122	0.021
	343.15	2.830	2.027	1.379	−2.368	0.027
	353.15	3.255	3.002	1.379	−3.819	0.031
$\Delta\eta$/mPa·s	303.15	−97.24	59.41	−56.88	50.56	0.112
	308.15	−73.09	44.90	−41.53	35.08	0.090
	313.15	−55.87	34.06	−33.47	29.12	0.082
	318.15	−43.94	25.97	−22.67	19.60	0.060
	323.15	−34.79	21.71	−17.12	12.32	0.054
	333.15	−22.21	16.86	−9.560	1.186	0.038
	343.15	−15.00	13.23	−5.628	−2.799	0.027
	353.15	−10.11	11.12	−3.642	−5.493	0.023

Estimated coefficients and standard error values are also presented. The results show that water content has stronger effects on the physical properties and excess thermodynamic properties of ILs for the binary system of H_2O + [BMIM][BF_4]. The volumetric and transport properties of the hydrophilic IL can be significantly varied by adding water or changing temperature to meet the needs of industrial requirement.

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