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Density, Excess Molar Volume and Conductivity of Binary Mixtures of the Ionic Liquid 1,2-Dimethyl-3-hexylimidazolium Bis(trifluoromethylsulfonyl)imide and Dimethyl Carbonate

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The densities and conductivities of the binary mixtures of 1,2-dimethyl-3-hexylimidazolium bis(trifluoromethylsulfonyl)imide ([DMHIM][Tf_2N]) and dimethyl carbonate (DMC) were measured at temperatures of (293.15 to 333.15) K. The densities increase with an increase in the concentration of [DMHIM][Tf_2N] or a decrease in the temperature. The excess molar volumes V^E were calculated and fitted to the Redlich−Kister equation. The conductivity increases sharply to a maximum value when the concentration of [DMHIM][Tf_2N] is increased and decreases quickly after the maximum. The data obtained will be helpful for the application of the ionic liquids as electrolytes and also useful for the ionic liquids database.

Introduction

Ionic liquids (ILs), which consist of organic cations and weakly coordinating inorganic/organic anions, offer an opportunity for solving the safety problem of lithium-ion batteries (LBs)[1,2] due to its unique features such as negligible vapor pressure, good thermal stability, wide potential window, and large liquids range, et al.[3,4] For example, 1,2,3-trialkylsubstituted imidazolium based ILs have proven to work well in many batteries.[5−7]

However, the high viscosity of the pure ILs (usually (50 to 500) mPa·s) was often a bottleneck for their industrial application as electrolytes in LBs. To solve this problem, an effort was made to use the mixtures of ILs and organic carbonates, which can decrease the viscosity of the system to meet the practical requirements of industry.[1] To our knowledge, 1,2-dimethyl-3-hexylimidazolium bis(trifluoromethylsulfonyl)imide ([DMHIM][Tf_2N]) as electrolyte performs a good charge−discharge cycle in LBs.[5−9] Dimethyl carbonate (DMC) is a widely used organic carbonate with low viscosity due to the aliphatic structure.[10] However, no report was found about the properties of the mixtures of [DMHIM][Tf_2N] and DMC. For the purpose of LB engineering, it is necessary to know the thermodynamic properties (especially volumetric property) and electrochemical properties (conductivity) of the electrolytes. So, in this work, the density and conductivity of the [DMHIM][Tf_2N] and its mixtures with DMC were experimentally measured at temperatures of (293.15 to 333.15) K. The excess molar volume of the binary systems was also investigated and fitted to the Redlich−Kister equation.

Experimental Section

Materials. DMC was supplied by Alfa Aesar Co., Ltd. with a mass fraction >0.99. [DMHIM][Tf_2N] was prepared according to a modified procedure of previous reports.[5,7,9] The synthesized ILs were washed with distilled water until no bromide traces were detected by addition of 0.1 $mol\cdot L^{-1}$ $AgNO_3$ solution. Then the ILs was dried at 333 K under vacuum until the mass fraction of water below 1×10^{-4}, which was determined by Karl Fischer titration (751 GPD Titrino, Metrohm, Switzerland). ^1H NMR spectra of the ILs were determined by a Bruker Advance-600 spectrometer. The chemical shifts (DMSO, δ/ppm relative to TMS) appear as follows: 7.64 (d, 1H, $J = 2.04$ Hz), 7.60 (d, 1H, $J = 2.04$ Hz), 4.10 (t, 2H, $J = 15.12$ Hz), 3.74 (s, 3H), 2.57 (s, 3H), 1.70 (t, 2H, $J = 13.08$ Hz), 1.20 (m, 6H, $J = 13.74$ Hz), 0.80 (t, 3H, $J = 13.02$ Hz). The chemical shift of the peaks corresponded to the structure of [DMHIM][Tf_2N], and no impurity peaks were observed in the ^1H NMR spectrum.

Apparatus and Procedure. The mixtures of [DMHIM][Tf_2N] and DMC were prepared by mass using a BS124S electronic digital balance accurate to within ±0.1 mg. It was conducted in a glovebox ($[O_2] < 1 \times 10^{-6}$, $[H_2O] < 1 \times 10^{-6}$) filled with argon atmosphere at ambient temperature. The densities of the pure compounds and their mixtures were measured by the Anton Paar DMA 5000 vibrating-tube densimeter with an uncertainty of 10^{-5} $g\cdot cm^{-3}$ and accuracy of $\pm 5 \times 10^{-6}$ $g\cdot cm^{-3}$. The highest temperature accuracy (±0.001 K) is controlled traceably to national standards by two integrated Pt 100 platinum thermometers. The detailed calibrating and measuring step was the same as in our previous work.[11]

The conductivities of the mixture were measured by a DDS-307 conductivity meter (Shanghai Precision & Scientific Instrument Co., LTD, accurate to ±1.0 %) in the glovebox described above, the measuring temperature range was (298.15 to 333.15) K. The constant for the cell was determined by calibration before and after each sample measurement with 0.1 $mol\cdot L^{-1}$ KCl solution at 298.15 K, and the constant was (1.021 ± 0.002) cm^{-1}. The conductivity of 0.01 $mol\cdot L^{-1}$ KCl solution at 298.15 K was 1.409 $mS\cdot cm^{-1}$. The electrode probe was composed of two platinized platinum and separated by a fix distance, next it was submerged in the sample which is connected to an oil bath whose temperature was controlled, and then a precision thermometer was used to determine the temperature with an uncertainty of 0.01 K. The conductivity of the pure DMC was below 1 $\mu S\cdot cm^{-1}$ in the temperature range measured, which

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Table 1. Comparison of Experimental and Literature Values of Densities ρ ($g\cdot cm^{-3}$) of DMC and [DMHIM][Tf$_2$N] at Different Temperatures

T	DMC		[DMHIM][Tf$_2$N]	
K	exp.	lit.	exp.	lit.
293.15	1.069923	1.06954[10] 1.0699[12] 1.0700[13] 1.06969[14]	1.358188	
298.15	1.063337	1.06295[10] 1.0633[12] 1.0635[13] 1.06311[14] 1.06338[16] 1.0632[17] 1.06333[18]	1.353111	1.36[15]
303.15	1.056719	1.05635[10] 1.0567[12] 1.0565[13] 1.05671[18]	1.348707	
308.15	1.050071	1.05004[16] 1.0508[17] 1.05007[18]	1.344292	
313.15	1.043388	1.04301[10] 1.0434[12] 1.04310[14] 1.04339[18]	1.339879	1.33[15]
318.15	1.036687	1.03670[16] 1.0377[17]	1.335468	
323.15	1.029939	1.02959[10] 1.02994[16]	1.331055	
328.15	1.023246	1.02224[19]	1.326650	
333.15	1.016319	1.01600[10]	1.322240	1.30[15]

was out of the detection limit of the conductivity meter. It is so low that in this work the conductivity of the pure DMC was set as 0 $mS\cdot cm^{-1}$. Here, each measuring was repeated three times for all samples, and the results were averaged.

As the conductivities of pure [DMHIM][Tf$_2$N] are not available in any reported literatures, we only compared the density of the [DMHIM][Tf$_2$N] and DMC with present literature data (Table 1). It can be observed that the density values agree well with literature values.

Results and Discussion

The experimental data of density of the [DMHIM][Tf$_2$N] (1) + DMC (2) system are given in Table 2. The densities of the binary system versus mole fraction of [DMHIM][Tf$_2$N] (x_1) at the temperature of (293.15 to 333.15) K are presented in Figure 1. It can be observed that the densities increase with the increasing of [DMHIM][Tf$_2$N] content or the decreasing of temperature.

The excess molar volumes (V^E) were calculated from the experimental data by eq 1.[20,21]

$$V^E = \frac{x_1 M_1 + x_2 M_2}{\rho} - \left(\frac{x_1 M_1}{\rho_1} + \frac{x_2 M_2}{\rho_2}\right) \qquad (1)$$

where ρ represents the density of mixtures, x_1 and x_2 are the mole fractions, M_1 and M_2 are molar masses, and ρ_1 and ρ_2 are densities of [DMHIM][Tf$_2$N] (1) and DMC (2), respectively. All of the values of excess molar volume were fitted by the Redlich–Kister polynomial eq 2.[20,21]

$$V^E = x_1(1 - x_1)\sum_{i=0}^{p} A_i(2x_1 - 1)^i \qquad (2)$$

Table 2. Experimental Densities, ρ, Excess Molar Volume, V^E, and Conductivity, κ, of [DMHIM][Tf$_2$N] (1) + DMC (2)

x_1	T = 293.15 K	T = 298.15 K	T = 303.15 K	T = 308.15 K	T = 313.15 K	T = 318.15 K	T = 323.15 K	T = 328.15 K	T = 333.15 K
					$\rho/g\cdot cm^{-3}$				
0.0000	1.069923	1.063337	1.056719	1.050071	1.043388	1.036687	1.029939	1.023246	1.016319
0.0466	1.120021	1.113880	1.107709	1.101519	1.096103	1.089110	1.082852	1.076680	1.070279
0.0774	1.145967	1.140013	1.134134	1.128205	1.122998	1.116298	1.110319	1.104425	1.098507
0.1154	1.173259	1.167458	1.161839	1.156118	1.151087	1.144641	1.138890	1.133315	1.127614
0.1633	1.201700	1.195922	1.190526	1.185040	1.180199	1.174051	1.168728	1.163271	1.157769
0.2269	1.231144	1.225504	1.220217	1.214852	1.210160	1.204452	1.199159	1.193979	1.188579
0.3131	1.261370	1.255942	1.250930	1.245851	1.241279	1.235847	1.230719	1.225631	1.220429
0.4385	1.292476	1.287184	1.282322	1.277481	1.272945	1.267890	1.263009	1.258144	1.253164
0.6368	1.324480	1.319317	1.314702	1.310137	1.305673	1.300910	1.296312	1.291790	1.287102
0.7472	1.337221	1.332182	1.327652	1.323097	1.318619	1.314090	1.309514	1.304969	1.300467
1.0000	1.358188	1.353111	1.348707	1.344292	1.339879	1.335468	1.331055	1.326650	1.322240
					$V_E/cm^3\cdot mol^{-1}$				
0.0466	−0.2236	−0.2516	−0.2722	−0.2943	−0.3103	−0.3451	−0.3715	−0.402	−0.4317
0.0774	−0.2843	−0.3226	−0.3580	−0.3922	−0.4220	−0.4662	−0.5069	−0.5526	−0.6164
0.1154	−0.3795	−0.4249	−0.4720	−0.5129	−0.5537	−0.6014	−0.6508	−0.7155	−0.7872
0.1633	−0.4835	−0.5218	−0.5761	−0.6248	−0.6780	−0.7317	−0.8092	−0.8706	−0.946
0.2269	−0.5682	−0.6089	−0.6553	−0.6965	−0.7575	−0.8282	−0.8894	−0.9621	−1.0258
0.3131	−0.6122	−0.6625	−0.7173	−0.7672	−0.8318	−0.8974	−0.9514	−1.0087	−1.0664
0.4385	−0.5654	−0.6126	−0.6538	−0.7021	−0.7545	−0.8209	−0.8719	−0.9236	−0.9703
0.6368	−0.3706	−0.4069	−0.4398	−0.4859	−0.5232	−0.5645	−0.6104	−0.6693	−0.7052
0.7472	−0.2738	−0.3185	−0.3432	−0.3661	−0.3855	−0.4280	−0.4592	−0.4863	−0.5297
					$\kappa/mS\cdot cm^{-1}$				
0.0000		0	0	0	0	0	0	0	0
0.0466		3.401	3.780	4.160	4.541	4.921	5.301	5.681	6.061
0.0774		5.792	6.487	7.182	7.877	8.572	9.267	9.962	10.66
0.1154		7.552	8.198	8.842	9.487	10.13	10.77	11.42	12.07
0.1633		8.390	8.931	9.472	10.01	10.55	11.09	11.63	12.17
0.2269		8.134	8.618	9.101	9.585	10.07	10.55	11.04	11.52
0.3131		6.310	6.889	7.510	8.121	8.760	9.330	9.979	10.67
0.4385		5.090	5.530	6.099	6.669	7.060	7.821	8.340	9.150
0.6368		2.301	2.961	3.610	4.331	4.921	5.482	6.231	6.860
0.7472		1.640	2.091	2.751	3.499	3.990	4.690	5.369	6.050
1.0000		1.080	1.577	2.134	2.710	3.191	3.689	4.260	4.801

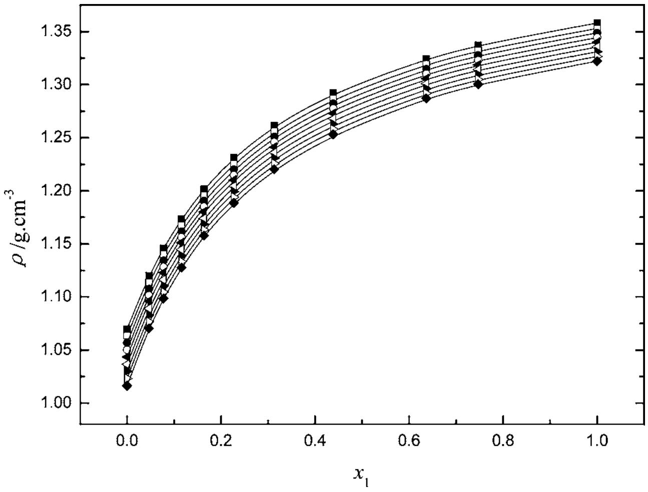

Figure 1. Density vs mole fraction x_1 in [DMHIM][Tf$_2$N] (1) + DMC (2): ■, 293.15 K; □, 298.15 K; ●, 303.15 K; ○, 308.15 K; solid left pointing traingle, 313.15 K; open left pointing triangle, 318.15 K; solid right pointing triangle, 323.15 K; open right pointing triangle, 328.15 K; and ♦, 333.15 K.

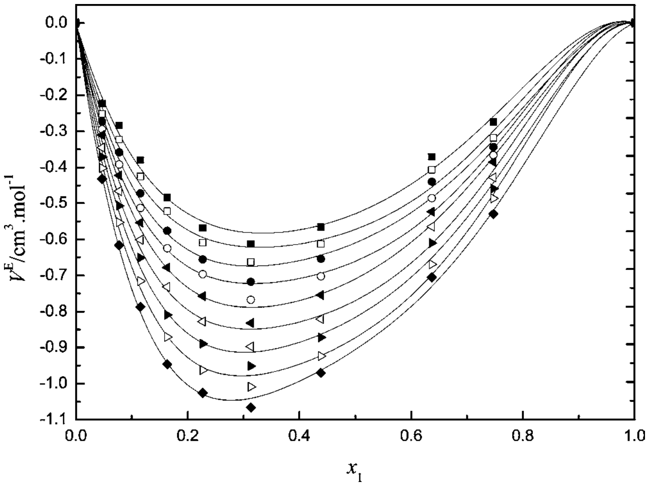

Figure 2. Excess molar volume V^E vs mole fraction x_1 in [DMHIM][Tf$_2$N] (1) + DMC (2): ■, 293.15 K; □, 298.15 K; ●, 303.15 K; ○, 308.15 K; solid left pointing triangle, 313.15 K; open left pointing triangle, 318.15 K; solid right pointing triangle, 323.15 K; open right pointing triangle, 328.15 K; and ♦, 333.15 K. The solid curves are calculated with the Redlich−Kister equation, and the symbols represent experimental values.

Table 3. Coefficients of the Redlich−Kister Equation for V^E of [DMHIM][Tf$_2$N] (1) + DMC (2)

T/K	A_0	A_1	A_2	A_3	σ
293.15	−2.1149	1.2575	−0.2646	1.5630	0.0207
298.15	−2.2893	1.1442	−0.4492	2.0869	0.0250
303.15	−2.4389	1.3038	−0.7210	2.0711	0.0233
308.15	−2.6423	1.3130	−0.6649	2.5492	0.0225
313.15	−2.8380	1.5962	−0.7973	2.3226	0.0203
318.15	−3.0786	1.5923	−0.8819	2.8738	0.0257
323.15	−3.2644	1.7654	−1.2172	2.8675	0.0221
328.15	−3.4911	1.8544	−1.3540	3.2771	0.0173
333.15	−3.6231	2.0327	−2.0957	3.1502	0.0136

where A_i are adjustable parameters. The standard derivation σ was correlated by eq 3.

$$\sigma = \left[\frac{\sum (V^E_{\mathrm{exp}} - V^E_{\mathrm{cal}})^2}{n - p}\right]^{1/2} \tag{3}$$

where n was the number of experimental data and p was the number of coefficients of the Redlich−Kister equation. The V^E are correlated by the three-term Redlich−Kister equation, namely $p = 3$. The data of excess molar volume were also presented in Table 2. The excess molar volume versus the mole fraction of [DMHIM][Tf$_2$N] was plotted in Figure 2. The values of the parameters A_i and standard deviations σ were listed in Table 3.

The excess molar volumes are negative over the entire composition range for the two binary systems, and the absolute values of excess molar volumes increase slightly with the an increase of the temperature from (293.15 to 333.15) K. A minimum V^E is located closely at $x_1 = 0.3$ where the quasi-clathrate is probably formed.[3,22] A similar phenomenon was observed in a series of works.[23−25] The negative excess molar volumes indicate that a more efficient packing and/or attractive interaction occurs when [DMHIM][Tf$_2$N] and DMC are mixed, which is favorable for the application of the mixtures as base electrolyte in electronic devices.

The experimental data of conductivity of the binary system are also given in Table 2 and are plotted against the x_1 at different temperatures in Figure 3. The conductivities of the mixtures increase sharply in the dilute solutions and then decrease after reaching a maximum value at about $x_1 = 0.1633$. This phenomenon exhibits a typical aggregation behavior of the classical properties of ILs and solvent mixed solutions.[26−28] It is reported that imidazolium cations will aggregate as the forms of A_nB_{n-1} and A_nB_{n+1} in solutions.[29] The aggregation brings three effects: (i) an increase in the viscosity and reduction in the mobility of the charge carriers, (ii) a reduction of the number of charge carriers and a promotion of the dissociation of lithium salt, and (iii) the restriction of molecular motion and a weakening of migrating competition among lithium ions.[29] From this point of view, aggregation, in the case of no serious influence to the conductivity, may improve the performance of the electrolyte solution. It is suggested that the mole fraction of $x_1 = (0.11−0.16)$ is a superior choice for the mixtures to be used as the base electrolyte. Otherwise, it can be observed that the curvature becomes larger as the temperature increases. The higher temperature may weaken the influence of aggregation to the conductivity, which suggests that a higher temperature

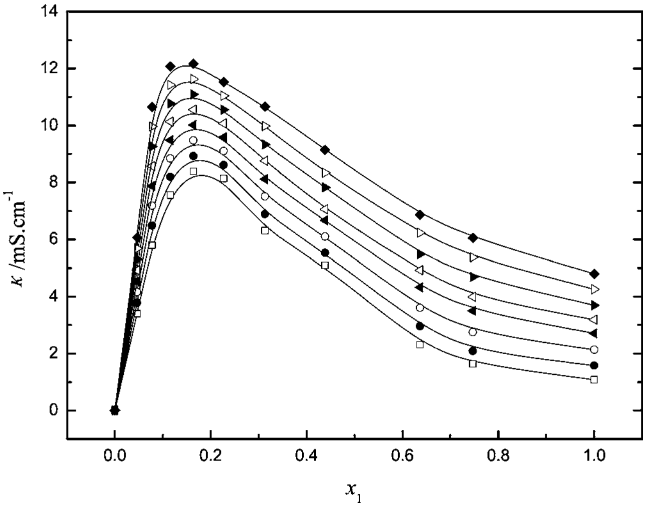

Figure 3. Conductivity vs mole fraction x_1 in [DMHIM][Tf$_2$N] (1) + DMC (2): □, 298.15 K; ●, 303.15 K; ○, 308.15 K; solid left pointing triangle, 313.15 K; open left pointing triangle, 318.15 K; solid right pointing triangle, 323.15 K; open right pointing triangle, 328.15 K; and ♦, 333.15 K.

is better for the ILs to act as base electrolytes due to its relatively higher conductivity.

Conclusion

The densities and conductivities of the binary system of [DMHIM][Tf_2N] and DMC were measured in the temperature range of (293.15 to 333.15) K. The density of the mixtures increases with an increase in the mole fraction of [DMHIM][Tf_2N] or with a decrease in the temperature. The excess molar volumes of the systems were obtained from experimental data and fitted to the Redlich−Kister equation. The conductivity of the mixtures increases sharply with an increase in the mole fraction of [DMHIM][Tf_2N] in the dilute solutions and then decreases after reaching a maximum value. Aggregation of the ILs was observed in the binary mixture, and it may be good for the solutions to act as the base electrolyte with a proper degree.

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