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Densities and Viscosities for Binary Mixtures of the Ionic Liquid *N*-Ethyl Piperazinium Propionate with *n*-Alcohols at Several Temperatures

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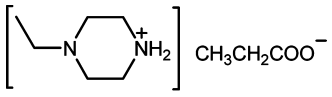
Supporting Information

ABSTRACT: A novel ionic liquid *N*-ethyl piperazinium propionate, [NEPP], was prepared, and the densities and viscosities for the binary mixtures of [NEPP] with methanol, ethanol, *n*-propanol, and *n*-butanol were measured over the whole concentration range at (298.15, 303.15, 308.15, and 313.15) K and 0.1 MPa. The data of the excess molar volume, V_m^E, were calculated and fitted with the Redlich−Kister type polynomial equation. The values of V_m^E of the investigated systems are all negative, indicating that the ion−dipole interactions play important roles between the molecules of the ionic liquid and the alcohols.

INTRODUCTION

Ionic liquids (ILs) completely composed of ions have many unique physicochemical properties, such as a broad liquid range, negligible vapor pressure, and high thermal stability.[1−4] They have been widely used as green solvents replacing traditional organic solvents in separation science.[5,6] In our laboratory, a series of novel *N*-alkyl piperazinium-based ILs have been prepared, and some of them have been used as extractants for removing aromatics and sulfides from hydrocarbon fuels. *N*-Ethyl piperazinium propionate, [NEPP], shown in Figure 1, is found to exhibit excellent extraction performance.



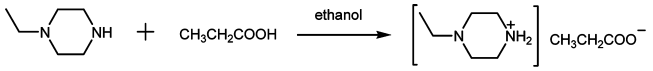
[NEPP]

Figure 1. Structures of *N*-ethyl piperazinium propionate, [NEPP].

However, the high viscosity of [NEPP] leads to difficulties in the practical process of extraction. With reference to many ILs introduced in literature,[7−9] this problem could be resolved partially by means of adding organic solvents to [NEPP]. Therefore, the thermodynamic and transport properties of [NEPP] with molecular solvents are of great importance in actual operation. Knowledge of the temperature and composition dependence of the density and viscosity is especially fundamental for the IL [NEPP] to be effectively used as a potential solvent in extraction.[10,11]

This paper reports the densities and viscosities for binary mixtures of [NEPP] with methanol, ethanol, *n*-propanol, and *n*-butanol over the entire range of mole fraction at (298.15, 303.15, 308.15, and 313.15) K and atmospheric pressure. The excess molar volume is calculated and discussed concerning the possible influence of molecular interactions.

Scheme 1. Synthesis of *N*-Ethyl Piperazinium Propionate [NEPP]



1 2 [NEPP]

Table 1. Dielectric Constant, ε, Density, ρ, and Viscosity, η, with Literature Data for Pure Components at T = 298.15 K

		ρ (g·cm^{-3})		η (mPa·s)	
compound	ε	exptl	lit.	exptl	lit.
[NEPP]	N/A	1.00836	N/A	243.5	N/A
methanol	32.62[18]	0.78669	0.7865[12]	0.5580	0.5768[12]
			0.78648[13]		0.5514[13]
			0.78664[14]		0.577[14]
			0.78677[15]		
ethanol	24.33[18]	0.78550	0.7855[12]	1.091	1.0961[12]
			0.78506[13]		1.078[13]
			0.78517[14]		1.09[14]
n-propanol	20.45[18]	0.79956	0.7994[12]	1.941	1.9468[12]
			0.79974[13]		1.9448[13]
			0.79952[14]		1.94[14]
n-butanol	17.55[18]	0.80581	0.80614[13]	2.577	2.5723[13]
			0.80578[16]		2.52[16]
			0.80580[17]		2.587[17]

EXPERIMENTAL SECTION

Chemicals. *N*-Ethyl piperazine, propionic acid, and *n*-propanol were supplied by Aladdin Chemistry Co. Ltd., Shanghai, China, with the mass fraction purity higher than

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Table 2. Experimental Densities, ρ, and Molar Excess Volumes, V_m^E, for Binary Mixtures of Alcohols (1) + [NEPP] (2)

	ρ (g·cm^{-3})				V_m^E (cm^3·mol^{-1})			
x_1	298.15 K	303.15 K	308.15 K	313.15 K	298.15 K	303.15 K	308.15 K	313.15 K
Methanol (1) + [NEPP] (2)								
0.0000	1.00836	1.00438	1.00033	0.99624	0.0000	0.0000	0.0000	0.0000
0.1070	1.00494	1.00096	0.99692	0.99286	−0.3790	−0.3862	−0.3975	−0.4102
0.2100	1.00073	0.99674	0.99272	0.98868	−0.7050	−0.7186	−0.7406	−0.7639
0.2970	0.99626	0.99230	0.98829	0.98426	−0.9514	−0.9748	−1.0036	−1.0334
0.4020	0.98936	0.98541	0.98141	0.97738	−1.2098	−1.2415	−1.2779	−1.3151
0.5021	0.98055	0.97659	0.97258	0.96855	−1.4062	−1.4424	−1.4835	−1.5256
0.5994	0.96897	0.96499	0.96097	0.95691	−1.5519	−1.5918	−1.6356	−1.6803
0.7015	0.95145	0.94738	0.94330	0.93919	−1.6137	−1.6508	−1.6938	−1.7379
0.7998	0.92533	0.92115	0.91696	0.91273	−1.5274	−1.5605	−1.5983	−1.6372
0.8999	0.87978	0.87541	0.87101	0.86657	−1.1455	−1.1692	−1.1948	−1.2212
1.0000	0.78669	0.78199	0.77725	0.77248	0.0000	0.0000	0.0000	0.0000
Ethanol (1) + [NEPP] (2)								
0.0000	1.00836	1.00438	1.00033	0.99624	0.0000	0.0000	0.0000	0.0000
0.0971	1.00279	0.99881	0.99477	0.99070	−0.2978	−0.3034	−0.3121	−0.3209
0.2017	0.99531	0.99132	0.98729	0.98323	−0.5404	−0.5504	−0.5666	−0.5834
0.3033	0.98658	0.98260	0.97858	0.97453	−0.7534	−0.7707	−0.7935	−0.8168
0.3983	0.97663	0.97266	0.96865	0.96459	−0.9228	−0.9461	−0.9740	−1.0018
0.5001	0.96341	0.95943	0.95541	0.95135	−1.0618	−1.0883	−1.1191	−1.1505
0.6006	0.94674	0.94275	0.93870	0.93462	−1.1466	−1.1752	−1.2068	−1.2392
0.6994	0.92528	0.92123	0.91715	0.91303	−1.1569	−1.1834	−1.2134	−1.2440
0.8001	0.89528	0.89116	0.88701	0.88282	−1.0419	−1.0635	−1.0880	−1.1128
0.8998	0.85247	0.84824	0.84398	0.83968	−0.7264	−0.7389	−0.7528	−0.7669
1.0000	0.78550	0.78120	0.77685	0.77247	0.0000	0.0000	0.0000	0.0000
n-Propanol (1) + [NEPP] (2)								
0.0000	1.00836	1.00438	1.00033	0.99624	0.0000	0.0000	0.0000	0.0000
0.1036	1.00019	0.99623	0.99223	0.98820	−0.1959	−0.2021	−0.2148	−0.2284
0.2026	0.99126	0.98730	0.98330	0.97927	−0.3764	−0.3861	−0.4015	−0.4185
0.3041	0.98078	0.97682	0.97283	0.96880	−0.5710	−0.5843	−0.6037	−0.6236
0.4017	0.96885	0.96494	0.96099	0.95699	−0.7201	−0.7430	−0.7708	−0.7970
0.4993	0.95501	0.95107	0.94708	0.94306	−0.8886	−0.9090	−0.9326	−0.9572
0.5997	0.93704	0.93309	0.92909	0.92505	−0.9238	−0.9440	−0.9666	−0.9898
0.6994	0.91503	0.91105	0.90703	0.90297	−0.9103	−0.9269	−0.9462	−0.9662
0.8009	0.88657	0.88255	0.87849	0.87439	−0.8023	−0.8138	−0.8272	−0.8406
0.8997	0.85011	0.84604	0.84193	0.83778	−0.5359	−0.5385	−0.5434	−0.5481
1.0000	0.79956	0.79554	0.79148	0.78737	0.0000	0.0000	0.0000	0.0000
n-Butanol (1) + [NEPP] (2)								
0.0000	1.00836	1.00438	1.00033	0.99624	0.0000	0.0000	0.0000	0.0000
0.1025	0.99847	0.99450	0.99048	0.98643	−0.1586	−0.1609	−0.1687	−0.1766
0.2018	0.98750	0.98354	0.97953	0.97548	−0.2663	−0.2707	−0.2800	−0.2894
0.2999	0.97537	0.97143	0.96744	0.96341	−0.3732	−0.3818	−0.3940	−0.4059
0.3989	0.96140	0.95748	0.95350	0.94949	−0.4565	−0.4673	−0.4816	−0.4954
0.5016	0.94478	0.94087	0.93691	0.93291	−0.5244	−0.5366	−0.5499	−0.5641
0.6004	0.92629	0.92239	0.91842	0.91442	−0.5730	−0.5840	−0.5963	−0.6084
0.7012	0.90396	0.90005	0.89608	0.89207	−0.5588	−0.5659	−0.5745	−0.5829
0.8008	0.87776	0.87384	0.86987	0.86585	−0.5046	−0.5070	−0.5104	−0.5135
0.8995	0.84596	0.84205	0.83808	0.83408	−0.3253	−0.3235	−0.3214	−0.3194
1.0000	0.80581	0.80200	0.79813	0.79423	0.0000	0.0000	0.0000	0.0000

0.999, 0.995, and 0.997, respectively. Methanol (0.995), absolute ethanol (0.997), and *n*-butanol (0.995) were obtained from Shanghai Chemical Corporation, China. All reagents were used without further purification.

Synthesis of ILs. *N*-Ethyl piperazinium propionate, [NEPP], was synthesized first in our laboratory by direct neutralization of *N*-ethyl piperazine (1) and propionic acid (2) (see Scheme 1). The mixture of *N*-ethyl piperazine (0.1 mol) with ethanol (100 mL) was loaded into a 250 mL three-neck flask in an ice−water bath. A solution of propionic acid (0.1 mol) in ethanol (50 mL) was dripped slowly into the flask, and the mixture was stirred for 30 min. The reaction lasted for another 5 h at room temperature. The total reaction process was performed under the protection of nitrogen gas. The solvent was then removed by rotary evaporation under vacuum. The product was recrystallized at −20 °C with *n*-pentane and ether mixture and then kept in a vacuum drying oven before use. [NEPP] is liquid at room temperature. The water content of [NEPP] analyzed by Karl Fisher titration (Mettler-Toledo DL32/DL39, Switzerland) was below 100 ppm.

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Table 3. Experimental Viscosities, η, for Binary Mixtures of Alcohols (1) + [NEPP] (2)

	η (mPa·s)			
x_1	298.15 K	303.15 K	308.15 K	313.15 K
Methanol (1) + [NEPP] (2)				
0.0000	243.5	164.8	113.4	80.93
0.1070	172.2	121.0	86.27	62.74
0.2100	123.1	89.92	65.93	49.08
0.2970	84.02	63.49	47.38	36.14
0.4020	52.96	40.86	31.63	24.89
0.5021	32.54	25.51	20.36	16.50
0.5994	18.41	15.03	12.40	10.40
0.7015	9.416	7.866	6.730	5.804
0.7998	4.331	3.807	3.366	2.989
0.8999	1.754	1.592	1.451	1.327
1.0000	0.5580	0.5261	0.4966	0.4702
Ethanol (1) + [NEPP] (2)				
0.0000	243.5	164.8	113.4	80.93
0.0971	170.1	119.4	85.41	61.23
0.2017	109.9	82.60	60.30	44.79
0.3033	75.85	55.81	42.03	32.10
0.3983	48.89	37.01	28.65	22.63
0.5001	29.59	23.34	18.57	15.06
0.6006	17.18	14.00	11.54	9.602
0.6994	9.403	7.923	6.735	5.766
0.8001	4.925	4.279	3.740	3.287
0.8998	2.437	2.175	1.947	1.749
1.0000	1.091	0.9986	0.9139	0.8387
n-Propanol (1) + [NEPP] (2)				
0.0000	243.5	164.8	113.4	80.93
0.1036	160.3	113.6	81.46	59.74
0.2026	107.7	76.59	56.22	42.19
0.3041	70.22	52.12	39.45	30.42
0.4017	47.45	36.21	28.19	22.22
0.4993	30.81	24.26	19.38	15.67
0.5997	18.94	15.39	12.60	10.42
0.6994	11.31	9.444	7.899	6.684
0.8009	6.389	5.454	4.689	4.059
0.8997	3.609	3.150	2.762	2.435
1.0000	1.941	1.728	1.545	1.385
n-Butanol (1) + [NEPP] (2)				
0.0000	243.5	164.8	113.4	80.93
0.1025	177.5	122.1	84.94	61.07
0.2018	133.8	91.02	63.82	46.53
0.2999	98.72	67.32	47.84	35.22
0.3989	73.16	50.02	35.72	26.48
0.5016	53.48	36.42	26.02	19.37
0.6004	38.82	26.35	18.80	14.05
0.7012	27.07	18.12	12.92	9.675
0.8008	17.51	11.84	8.502	6.434
0.8995	9.611	6.701	4.966	3.870
1.0000	2.577	2.272	2.010	1.786

The synthesized [NEPP] characterized by ^{1}H NMR spectrum was carried out on a Bruker DMX 500 MHz NMR spectrometer, using $CDCl_3$ as the solvent and tetramethylsilane (TMS) as the internal standard, with the chemical shifts δ_H of 8.31 (s, 2H, −NH, and −OH), 3.05 (t, 4H, N−CH_2−C), 2.60 (m, 4H, C−CH_2−N), 2.45 (q, 2H, −CH_2−N), 2.21 (q, 2H, −CH_2−COO), and 1.09 (t, 6H, −CH_3). The purity of [NEPP] was higher than 98 % (mass fraction) according to the total peak integral in the ^{1}H NMR spectrum.

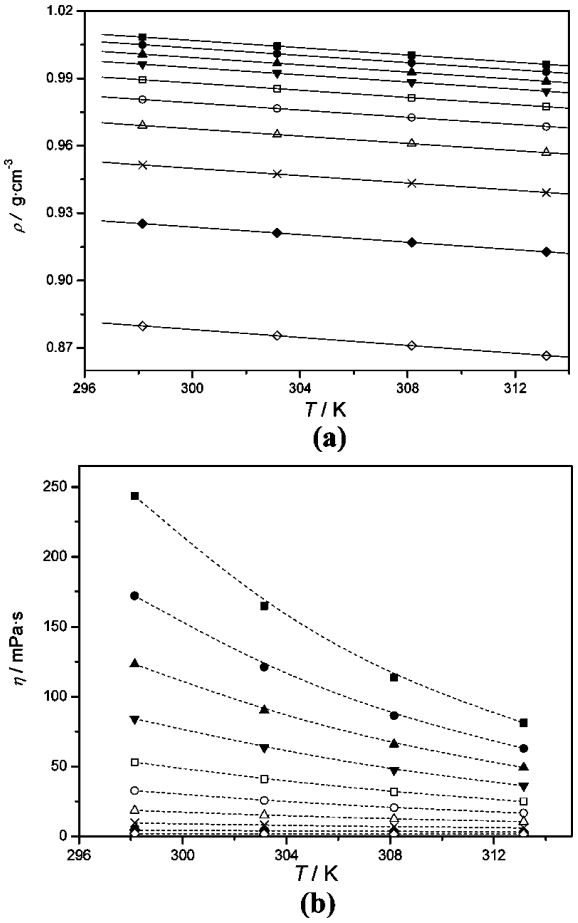

Figure 2. Densities (a) and viscosities (b) for the methanol (1) + [NEPP] (2) mixtures as a function of temperature with the mole fraction x_1: ■, 0.0000; ●, 0.1070; ▲, 0.2100; ▼, 0.2970; □, 0.4020; ○, 0.5021; △, 0.5994; ×, 0.7015; ♦, 0.7998; ◊, 0.8999.

Apparatus and Procedure. The binary mixtures were prepared in a 20 mL special stoppered glass vial by mass using an analytical balance (Mettler-Toledo AL204) with a stated precision of ± $1\cdot10^{-4}$ g. The uncertainty in mole fraction was ± $1\cdot10^{-4}$.

Density measurements of the binary mixtures at (298.15, 303.15, 308.15, and 313.15) K were carried out by using a DMA 5000 M density meter (Anton Paar). The density meter was calibrated with ultrapure water and dry air, and the uncertainty of the density measurement was ± $5\cdot10^{-6}$ g·cm^{-3}.

Viscosities were measured by AMVn viscometer (Anton Paar) at different temperatures and atmospheric pressure. The temperature was controlled by a built-in Peltier thermostat with a precision of ± 0.01 K. Three kinds of capillary/ball with different diameters (1.6 mm, 1.8 mm, and 4.0 mm) were selected to measure viscosities from (0.3 to 2500) mPa·s. The overall uncertainty of the viscosity measurements was within 0.5 %.

■ RESULTS AND DISCUSSION

The experimental density (ρ) and viscosity (η) data of pure components compared with the available literature data[12−18] are given in Table 1.

The measured densities and the dynamic viscosities for the binary systems of *n*-alcohol (1) + [NEPP] (2) at T = (298.15, 303.15, 308.15, and 313.15) K and atmospheric pressure over the whole composition range are given in Tables 2 and 3,

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Table 4. Results of the Redlich–Kister Correlation for Excess Molar Volume for *n*-Alcohol (1) + [NEPP] (2) Mixtures at Different Temperatures

T/K	A_0	A_1	A_2	A_3	A_4	σ
			Methanol (1) + [NEPP] (2)			
298.15	−5.6338	−3.3088	−2.5660	−3.3503	−2.5886	0.0076
303.15	−5.7830	−3.3883	−2.5022	−3.4100	−2.7360	0.0077
308.15	−5.9472	−3.4616	−2.5512	−3.4573	−2.7483	0.0079
313.15	−6.1153	−3.5358	−2.6055	−3.4965	−2.7735	0.0080
			Ethanol (1) + [NEPP] (2)			
298.15	−4.2580	−2.2697	−1.4092	−1.0146	−1.3799	0.0023
303.15	−4.3667	−2.3344	−1.3648	−0.9888	−1.4312	0.0026
308.15	−4.4901	−2.3831	−1.3791	−0.9674	−1.4173	0.0026
313.15	−4.6151	−2.4320	−1.4011	−0.9457	−1.3908	0.0026
			n-Propanol (1) + [NEPP] (2)			
298.15	−3.4663	−2.0222	−0.0862	−0.5913	−1.2403	0.0125
303.15	−3.5530	−2.0516	−0.0159	−0.5128	−1.2570	0.0115
308.15	−3.6549	−2.0638	0.0486	−0.4176	−1.3437	0.0105
313.15	−3.7565	−2.0797	0.0918	−0.3025	−1.4072	0.0103
			n-Butanol (1) + [NEPP] (2)			
298.15	−2.1042	−1.1955	−0.7596	0.0132	−0.1833	0.0061
303.15	−2.1532	−1.1991	−0.6811	0.0612	−0.1925	0.0063
308.15	−2.2093	−1.1987	−0.6346	0.1636	−0.2033	0.0068
313.15	−2.2659	−1.1973	−0.5753	0.2653	−0.2348	0.0071

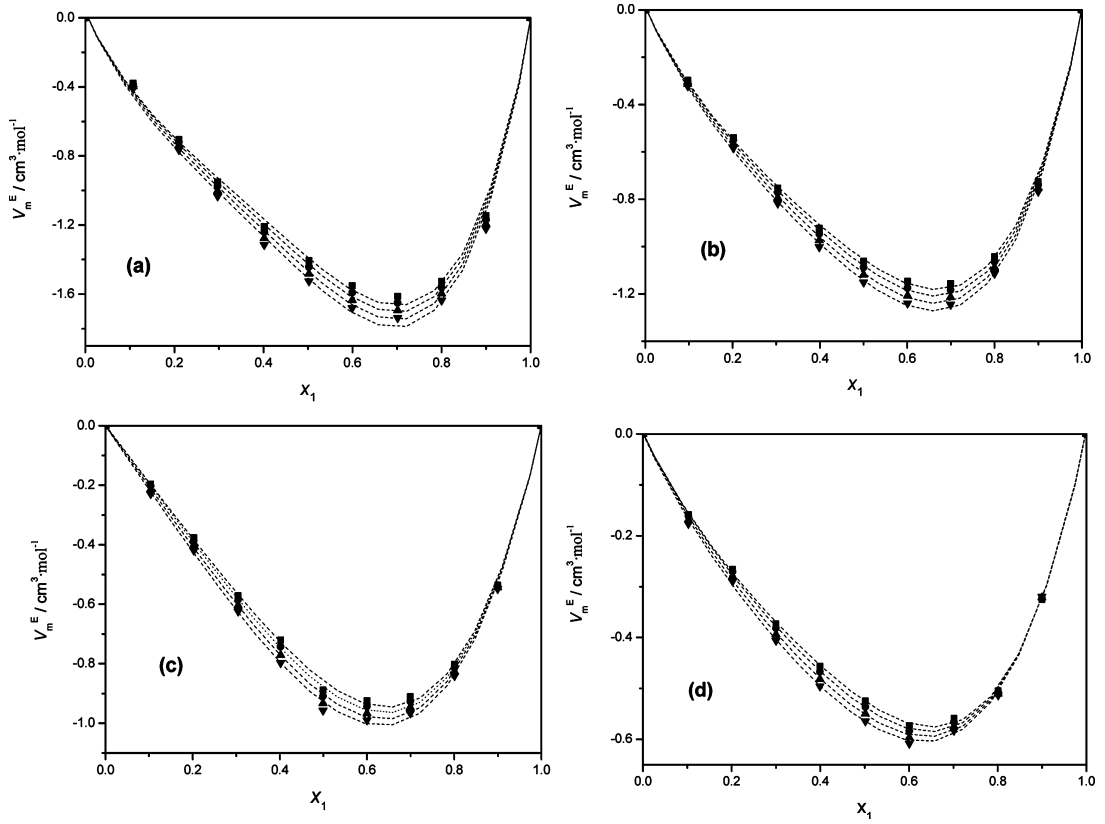

Figure 3. Excess molar volumes, V_m^E, for the systems: (a) methanol (1) + [NEPP] (2); (b) ethanol (1) + [NEPP] (2); (c) *n*-propanol (1) + [NEPP]; (d) *n*-butanol (1) + [NEPP] (2) at different temperatures: ■, 298.15 K; ●, 303.15 K; ▲, 308.15 K; ▼, 313.15 K. The dashed lines were calculated using the Redlich–Kister eq 2.

respectively. As can be seen, the densities and viscosities are much higher for the IL than for alcohols and decrease significantly with increasing mole fraction of alcohol or temperature. The temperature variation of densities and viscosities for [NEPP] and the binary mixtures is shown in Figure 2. A linear behavior for density versus temperature from Figure 2a and an exponential decay for viscosity versus temperature from Figure 2b are observed during the investigated temperature range, which are the same as those for many other ILs in literature.[10,19]

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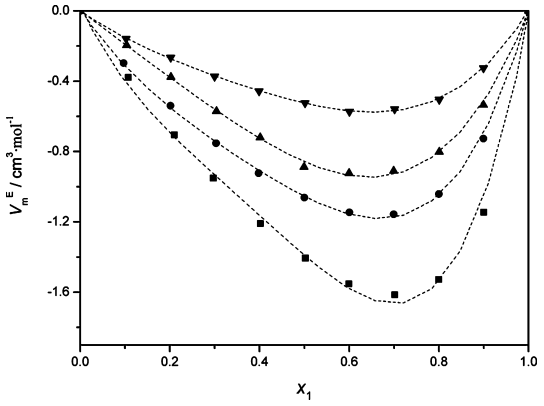

Figure 4. Excess molar volume versus composition for *n*-alconol (1) + [NEPP] (2) mixtures at 298.15 K: ■, methanol; ●, ethanol; ▲, *n*-propanol; ▼, *n*-butanol. The dash lines were calculated using the Redlich−Kister eq 2.

The excess molar volume, V_m^E, can be calculated by the following equations:

$$V_m^E = \frac{M_1x_1 + M_2x_2}{\rho_m} - \left(\frac{M_1x_1}{\rho_1} + \frac{M_2x_2}{\rho_2}\right) \quad (1)$$

where ρ_m is the density of the mixture and M_i, x_i, and ρ_i are the molecular mass, mole fraction, and density of the pure components *n*-alcohol (1) and [NEPP] (2), respectively. The values of V_m^E determined are listed in Table 2.

The values of V_m^E are correlated by the Redlich−Kister equation:

$$Y = x_1(1 - x_1)\sum_{i=1}^{k} A_i(x_1 - x_2)^{i-1} \quad (2)$$

where Y denotes V_m^E, A_i is the polynomial coefficient, and k is the degree of the polynomial expansion obtained by fitting the equation to the experimental values with a least-squares algorithm. The coefficients are summarized in Table 4, along with the standard deviations, σ, defined as follows:

$$\sigma = \sqrt{\frac{\sum_{i=1}^{N}(Y_{exp} - Y_{cal})_i^2}{N - k}} \quad (3)$$

where N is the number of experimental data and the subscript of exp and cal denotes experimental and calculated values, respectively.

Figure 3 gives the concentration dependence of the excess molar volume. As shown, the V_m^E values of the binary mixtures are negative in the whole concentration range, decreasing with the increase in alcohol concentration up to the mole fraction x_1 of 0.6 to 0.7, and then increasing with x_1. Much higher values of negative deviations from the ideality may be related to the strong ion−dipole interactions.[16,20] of the IL with alcohols. Figure 4 indicates that the magnitude of the negative V_m^E is sensitive to the alkyl length of the alcohol molecules. The absolute values of V_m^E at a given mole fraction in the binary mixtures follow the order: methanol > ethanol > *n*-propanol > *n*-butanol. Interestingly, it can be found that the order is the same as that of the dielectric constants of alcohols, which decreases with increasing the alkyl length (see Table 1). Therefore, the strength of ion−dipole interactions between the IL and the alcohols should be in agreement with this observed decreasing order.

CONCLUSIONS

The densities and the dynamic viscosities for binary mixtures of the IL, *N*-ethyl piperazinium propionate, [NEPP], with *n*-alcohols, methanol, ethanol, *n*-propanol, and *n*-butanol, have been reported as a function of temperature and at atmospheric pressure. The variation of density with temperature for the binary mixtures is represented as linear behavior. Excess molar volumes have been derived from the experimental data and fitted with the Redlich−Kister type polynomial equation. Very large negative values of excess molar volume over the whole composition range are observed for all of the investigated binary systems. The negative excess molar volumes indicate that the ion−dipole interactions play important roles between the molecules of the IL and the alcohols.

ASSOCIATED CONTENT

Supporting Information

^{1}H NMR of *N*-ethyl piperazinium propionate and materials. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

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