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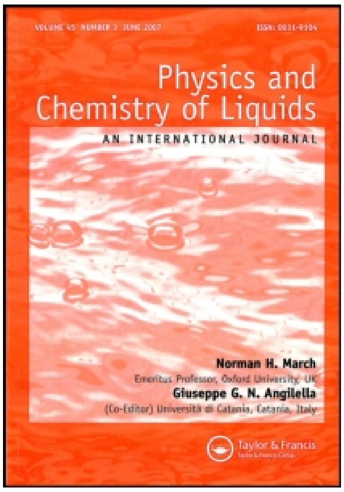

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Mutual solubilities of selected solvents and 1-(2-hydroxyethyl)-3-methylimidazolium tetrafluoroborate

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RESEARCH ARTICLE

Mutual solubilities of selected solvents and 1-(2-hydroxyethyl)-3-methylimidazolium tetrafluoroborate

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1-(2-Hydroxyethyl)-3-methylimidazolium tetrafluoroborate ([HYDEMIM][BF_4]) ionic liquid was characterised by infrared spectroscopy, nuclear magnetic resonance, ultraviolet-visible spectra and thermogravimetric analysis. Mutual mass fraction solubilities of the 12 selected solvents (ethanol, 1-propanol, 1-butanol, benzene, toluene, *o*-xylene, *m*-xylene, *p*-xylene, ethylbenzene, dichloromethane, chloroform and carbon tetrachloride) and [HYDEMIM][BF_4] in the temperature range from 283.16 to 353.61 K were measured using a cloud-point method. Measured solubility value S was correlated as a function of temperature by a second-order polynomial.

Keywords: solubility; 1-(2-hydroxyethyl)-3-methylimidazolium tetrafluoroborate; ionic liquid; organic solvent; measurement

1. Introduction

In the last decade ionic liquids (ILs) have attracted considerable interest both as environmentally benign media and because of their fascinating properties. They have been employed as solvents for many chemical processes, such as organometallic catalysis [1], electrochemistry [2], organic synthesis [3], etc.

Recently, in IL research more attention has been paid on the task-specific ionic liquids (TSILs) [4]. For example, hydroxyl-functionalised ILs, 1-(2-hydroxyethyl)-3-methylimidazolium tetrafluoroborate ([HYDEMIM][BF_4]) (its formula is shown in Figure 1) has been introduced widely as soluble support in the liquid-phase organic synthesis [5–7], which combined the advantages of ILs and heterogeneous support.

To aid the development of ILs for reactions and separations, a number of liquid–liquid phase equilibria containing ILs have been reported. Shang *et al.* [8] measured the solubilities of benzene, toluene and xylene isomers in 1-butyl-3-methylimidazolium tetrafluoroborate. Anthony *et al.* [9] systematically studied the solution thermodynamics of imidazolium-based ILs and water. Domańska *et al.* [10] determined the solubility of 1-alkyl-3-ethylimidazolium-based ILs in water and 1-octanol. Huo *et al.* [11] reported the solubility of alcohols and aromatic compounds in imidazolium-based ILs. Crosthwaite *et al.* [12] studied the liquid phase behaviour of imidazolium-based ILs with alcohols. Freire *et al.* [13] presented

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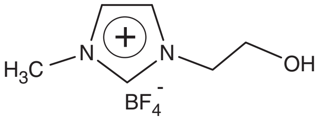

Figure 1. Structure of [HYDEMIM][BF_4].

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an overview of the mutual solubilities of water–imidazolium-based ILs systems. In this work, the mutual solubilities of binary systems: [HYDEMIM][BF_4] + alcohol (ethanol, 1-propanol or 1-butanol), benzene derivative (benzene, toluene, *o*-xylene, *m*-xylene, *p*-xylene or ethylbenzene) or chlorinated methane (dichloromethane, chloroform, carbon tetrachloride) were measured.

2. Experimental section

2.1. *Materials*

[HYDEMIM][BF_4] was purchased from Lanzhou Institute of Chemical Physics of the Chinese Academy of Sciences. Its mass fraction purity was above 99% according to specifications, with the following certified amounts of impurities: $w\,(Cl^-) < 5 \times 10^{-4}$. The mass fraction of water was checked to be less than 10^{-3} by Karl Fischer titration. All the other chemicals in the measurement were purchased from Sinopharm Chemical Reagent Beijing Co. Ltd., and their purities were as follows: ethanol (99.7%), 1-propanol (99.0%), 1-butanol (99.0%), benzene (99.5%), toluene (99.5%), *o*-xylene (98.5%), *m*-xylene (95.0%), *p*-xylene (95.0%), ethylbenzene (99.0%), dichloromethane (99.5%), chloroform (99.0%) and carbon tetrachloride (99.5%). They were used without further purification. The water content in each of the solvents was less than 0.20%.

2.2. *Apparatus*

IR spectra were measured with a Nicolet MAGNA 750 fitted with a Nic-plan IR microscope. ^1H NMR spectra were recorded on a Varian Unity Inova-400 spectrometer. The electronic spectra were recorded on a 756 PC UV/VIS spectrometer. Thermogravimetric analysis (TGA) was carried out with an SDT Q600 Simultaneous DTA-TGA thermogravimetric analyser.

A jacketed equilibrium cell with a working volume of 100 ml immersed in a circulating water bath with a thermostat (type 501, Shanghai Laboratory Instrument Works Co. Ltd.) with a precision of ±0.05 K as described by Shang *et al.* [8], an analytical balance (type TG328B, Shanghai Balance Instrument Works Co.) with an uncertainty of ±0.1 mg, a magnetic stirrer and a calibrated thermometer with a precision of 0.1 K were used in the experiment. The equilibrium cell was sealed to prevent the evaporation of solvent or solute during the experiment.

2.3. *Characterisation of [HYDEMIM][BF_4]*

^1H NMR (D_2O, 400 MHz): $\delta = 3.98$ (t, 3H, CH_3) (3.90) [14], 3.99–4.02 (dd, 2H, CH_2O) (3.85–3.88) [14], 4.37–4.40 (t, 2H, CH_2) (4.24) [14], 7.52 (d, 1H, CH=CH)

(7.44) [14], 7.57 (d, 1H, CH=CH) (7.49) [14], 8.77 (s, 1H, CH=N) (8.62) [14]. IR (cm^{-1}): 847 cm^{-1} (ν(C–N)) (847) [15], 1066 (ν(B–F)) (1063) [15], 1429 (ν(O–H)) (1430) [15], 1170 (ν(C–O)) (1170) [15], 1453, 1577 (ν(C=C, C=N)) (1452, 1577) [15], 2967, 2895 (ν(C=H)) (2966, 2892) [15], 3123, 3167 (ν(C=H)) (3122, 3165) [15], 3551 (ν(O–H)) (3554) [15]. UV-Vis (in C_2H_5OH): $\lambda_{\max}$ (nm) = 210.95 ($\varepsilon = 4109\ \mathrm{M}^{-1}\,\mathrm{cm}^{-1}$).

2.4. *Thermogravimetric analysis*

An SDT Q600 Simultaneous DTA-TGA thermogravimetric analyser was employed for TGA at a heating rate of 10 K min^{-1} under nitrogen from 296.73 to 1063.49 K. The initial decomposition temperature of [HYDEMIM][BF_4] was around 617.06 K, and the temperature at 95.40% mass loss was 674.99 K.

2.5. *Solubility measurement*

The solubilities were measured with the cloud-point method. For the measurement of the mass fraction solubility S of the 12 selected solvents in [HYDEMIM][BF_4] at each selected temperature, the amount of solute dissolved was determined by a gravimetric method. With a known mass of [HYDEMIM][BF_4] at a constant temperature, continuous stirring was applied, and the solute was added through a titrimeter (syringe type, 3 mm in diameter) until the solution became slightly turbid by careful observation. A 100 ml Erlenmeyer flask filled with a fixed mass of the solute and with the syringe inserted was weighed before and after the titration operation, and the difference was taken. The charging inlet was sealed with a rubber plug to prevent the solute from evaporating. The equilibrium temperature was taken from the calibrated thermometer that was inserted into the solution. The method of the solubility measurement of [HYDEMIM][BF_4] in the 12 selected solvents is similar to that described above. The amount of solvent used for each measurement was around 50 g. All of the measurements reported in this article were repeated three times at each temperature, and the average value was taken. The overall relative uncertainty of the mass fraction was about 3% at 310 K and improved further at higher temperatures because the solubility values at lower temperature were less than those at higher temperatures.

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3. Data correlation

The mutual mass fraction solubilities of the 12 selected solvents and [HYDEMIM][BF_4] are listed in Table 1. The mass fraction solubilities data were correlated as a function of temperature by the following form of a second-order polynomial:

$$100S = a + b(T) + c(T)^2, \tag{1}$$

where temperature is measured in kelvins. Parameters a, b and c correlated for the different systems and the standard deviations of the calculated value with the experimental data are listed in Table 2. The standard deviation, σ, of the experimental data from smoothed data is expressed as

$$\sigma = \sqrt{\frac{\sum(S_i^{\mathrm{exp}} - S_i^{\mathrm{calcd}})^2}{N-1}}, \tag{2}$$

Table 1. Measured mutual mass fraction solubilities (*S*) of the 12 selected solvents in [HYDEMIM][BF$_4$].

		Solubility of organic solvent in IL		Solubility of IL in organic solvent
Solvent	*T*(K)	100**S**	*T*(K)	100**S**
Ethanol	297.29	41.26	297.67	8.90
	298.31	44.37	298.49	9.79
	299.09	46.74	299.31	10.69
	300.42	50.77	300.69	12.48
	301.10	52.82	301.42	13.56
	302.32	56.48	302.65	15.58
	303.59	60.26	303.72	17.55
	304.89	64.12	304.99	20.14
	305.96	67.27	305.98	22.35
	306.67	69.36	306.86	24.45
	307.28	71.15	307.71	26.61
1-Propanol	296.98	35.89	296.45	7.315
	303.21	38.57	303.25	6.71
	305.96	42.48	305.87	7.67
	308.06	46.59	308.03	8.96
	310.88	53.64	310.72	11.20
	312.39	58.13	312.39	12.94
	313.35	61.25	313.24	13.93
	314.59	65.58	314.27	15.22
	315.21	67.88	315.34	16.68
	316.33	72.23	316.79	18.82
	316.89	74.51	317.43	19.83
	317.32	76.31	317.91	20.62
1-Butanol	298.03	23.15	298.23	2.58
	302.24	24.74	302.32	2.02
	305.52	26.86	305.39	2.17
	308.12	29.08	308.23	2.75
	312.94	34.47	312.44	4.40
	316.14	38.95	316.80	7.09
	319.18	43.88	319.56	9.31
	322.12	49.28	323.32	12.97
	325.90	57.12	326.17	16.25
	328.15	62.26	328.94	19.84
	329.67	65.94	330.63	22.23
	330.61	68.30	331.19	23.05
	331.19	69.79	331.44	23.43
	331.52	70.64	331.91	24.14
Benzene	313.42	0.068	314.89	0.101
	317.80	0.074	318.17	0.118
	322.94	0.084	322.80	0.142
	329.05	0.094	328.92	0.172
	333.43	0.103	334.49	0.199
	338.70	0.110	337.27	0.211
	342.49	0.113	343.46	0.238

(*Continued*)

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Table 1. Continued.

		Solubility of organic solvent in IL		Solubility of IL in organic solvent
Solvent	T(K)	100**S**	T(K)	100**S**
Toluene	308.83	0.105		
	312.42	0.111	312.90	0.135
	316.30	0.117	317.41	0.160
	321.70	0.126	321.36	0.182
	326.41	0.134	328.32	0.215
	332.82	0.145	332.44	0.240
	338.37	0.155	337.48	0.261
	343.24	0.165	343.31	0.273
o-Xylene	304.55	0.126	303.26	0.101
	310.17	0.148	312.45	0.181
	316.32	0.169	316.68	0.215
	322.49	0.189	322.50	0.258
	330.82	0.213	330.14	0.308
	339.57	0.233	339.38	0.361
	347.45	0.249	347.47	0.400
	353.22	0.258	353.39	0.424
m-Xylene	303.85	0.090	303.54	0.085
	309.24	0.100	311.28	0.119
	316.86	0.114	316.60	0.144
	322.28	0.126	322.21	0.172
	330.53	0.144	330.35	0.215
	339.29	0.165	339.67	0.266
	347.73	0.188	347.20	0.311
	353.61	0.205	353.34	0.349
p-Xylene	303.23	0.116	303.48	0.099
	309.47	0.134	311.22	0.124
	316.20	0.152	316.67	0.146
	323.52	0.170	323.37	0.177
	331.36	0.188	331.29	0.220
	339.16	0.204	339.82	0.273
	346.83	0.219	346.63	0.322
	353.21	0.230	353.06	0.372
Ethylbenzene	313.38	0.142	313.18	0.146
	317.82	0.153	318.87	0.179
	322.54	0.164	321.41	0.198
	326.43	0.172	326.30	0.225
	332.89	0.184	332.41	0.252
	338.76	0.193	338.37	0.284
	342.15	0.198	343.02	0.298
			345.48	0.306
Dichloromethane	283.16	22.48	283.42	0.055
	286.24	22.99	286.20	0.177
	291.38	23.86	291.44	0.396
	298.74	25.15	298.81	0.679
	302.65	25.85	302.50	0.809
	307.27	26.70	307.92	0.987
	313.43	27.87	313.53	1.155

(*Continued*)

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Table 1. Continued.

Solvent	T(K)	Solubility of organic solvent in IL 100S	T(K)	Solubility of IL in organic solvent 100S
Chloroform	288.42	12.34	295.73	0.0006
	298.24	13.12	303.62	0.0009
	307.96	13.78	310.61	0.0010
	317.73	14.32	319.48	0.0011
	327.11	14.72	323.14	0.0013
Carbon tetrachloride	287.65	0.547	287.63	0.153
	297.19	0.648	297.18	0.189
	300.15	0.677	300.17	0.205
	308.26	0.756	308.13	0.230
	315.10	0.818	315.38	0.246
	318.73	0.850	318.77	0.254

Table 2. Parameters in Equation (1) and standard deviations calculated from Equation (2) for each system.

System	a	b	$c \times 10^4$	σ
Ethanol + [HYDEMIM][BF_4]	−1459.27492	7.03626	−66.900	0.0033
1-Propanol +[HYDEMIM][BF_4]	9840.98807	−65.77592	1103.1	0.0073
1-Butanol + [HYDEMIM][BF_4]	3104.99714	−20.91188	354.70	0.0044
Benzene + [HYDEMIM][BF_4]	−1.6533	0.00903	−0.1129	0.0019
Toluene + [HYDEMIM][BF_4]	0.2110	−0.0022	0.0603	0.0016
o-Xylene + [HYDEMIM][BF_4]	−3.4279	0.0194	−0.2540	0.0024
m-Xylene + [HYDEMIM][BF_4]	0.6823	−0.0056	0.1203	0.0017
p-Xylene + [HYDEMIM][BF_4]	−2.0321	0.0112	−0.1360	0.0024
Ethylbenzene + [HYDEMIM][BF_4]	−3.1424	0.0183	−0.2494	0.0016
Dichloromethane + [HYDEMIM][BF_4]	14.4645	−0.1069	4.7750	0.0025
Chloroform + [HYDEMIM][BF_4]	−65.1154	0.4511	−6.3296	0.0021
Carbon tetrachloride + [HYDEMIM][BF_4]	−5.7159	0.0326	−0.3767	0.0024
[HYDEMIM][BF_4] + ethanol	7327.31398	−50.06592	856.00	0.0092
[HYDEMIM][BF_4] + 1-propanol	4381.2272	−29.09061	483.60	0.0036
[HYDEMIM][BF_4] + 1-butanol	2414.00916	−15.92628	262.90	0.0019
[HYDEMIM][BF_4] + benzene	−3.6757	0.0186	−0.2096	0.0024
[HYDEMIM][BF_4] + toluene	−7.6373	0.0432	−0.5862	0.0033
[HYDEMIM][BF_4] + *o*-xylene	−7.7194	0.0424	−0.5476	0.0018
[HYDEMIM][BF_4] + *m*-xylene	0.6786	−0.0082	0.2055	0.0022
[HYDEMIM][BF_4] + *p*-xylene	4.2061	−0.0299	0.5395	0.0023
[HYDEMIM][BF_4] + ethylbenzene	−7.0497	0.0393	−0.5196	0.0025
[HYDEMIM][BF_4] + dichloromethane	−34.4663	0.1989	−2.7200	0.0025
[HYDEMIM][BF_4] + chloroform	−0.0313	0.0002	−0.0026	0.0001
[HYDEMIM][BF_4] + carbon tetrachloride	−4.7403	0.0294	−0.4306	0.0023

where the superscript exp and calcd stand for experimental and calculated values, respectively, and N stands for the total number of experimental data points. Figures 2–7 represent the graphs of the mutual mass fraction solubilities of the 12 selected solvents and [HYDEMIM][BF_4] *versus* temperature.

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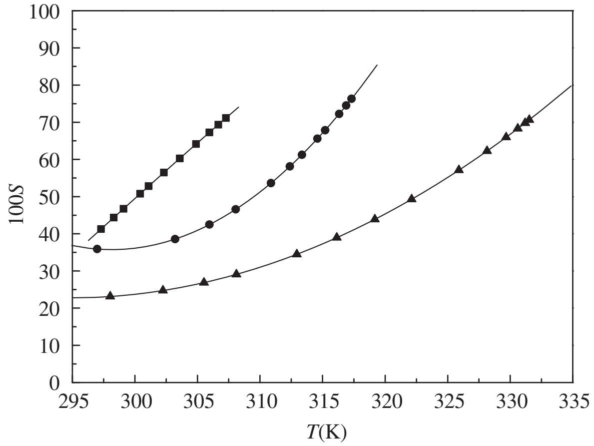

Figure 2. Mass fraction solubilities of ethanol, 1-propanol and 1-butanol in [HYDEMIM][BF$_4$]. Measured solubilities of ■, ethanol; ●, 1-propanol; ▲, 1-butanol in [HYDEMIM][BF$_4$] – calculated according to Equation (1).

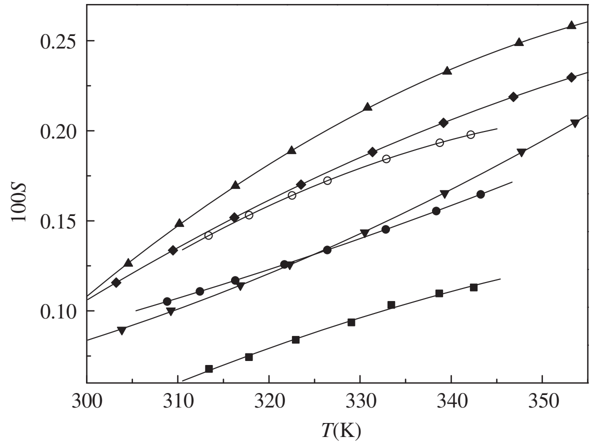

Figure 3. Mass fraction solubilities of benzene, toluene, *o*-xylene, *m*-xylene, *p*-xylene and ethylbenzene in [HYDEMIM][BF$_4$]. Measured solubilities of ■, benzene; ●, toluene; ▲, *o*-xylene; ▼, *m*-xylene; ♦, *p*-xylene; ○, ethylbenzene in [HYDEMIM][BF$_4$] – calculated according to Equation (1).

The separation factor, *SF*, is defined as

$$SF = \frac{S_i}{S_j}, \tag{3}$$

where S_i and S_j are the solubility of solutes *i* and *j* in ILs, respectively. For the xylene isomers studied, the values of S_i and S_j in [HYDEMIM][BF$_4$] or [BMIM][BF$_4$] for comparison and the values of separation factor, *SF*, [HYDEMIM][BF$_4$] or [BMIM][BF$_4$] for the separation of two xylene isomers mixture calculated using Equation (3) at $T = 298.15$ K are listed in Table 3.

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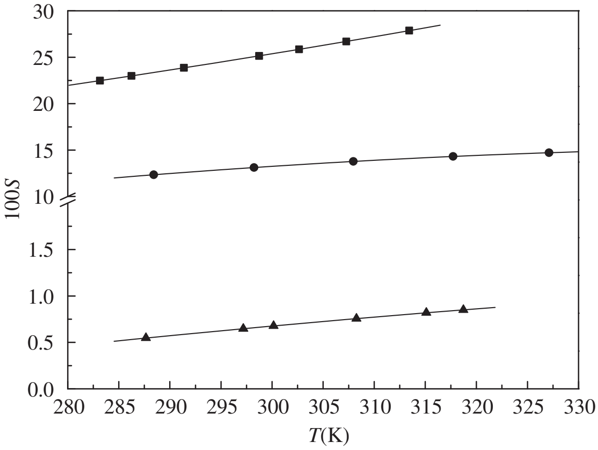

Figure 4. Mass fraction solubilities of dichloromethane, chloroform, carbon tetrachloride in [HYDEMIM][BF_4]. Measured solubilities of ■, dichloromethane; •, chloroform; ▲, carbon tetrachloride in [HYDEMIM][BF_4] – calculated according to Equation (1).

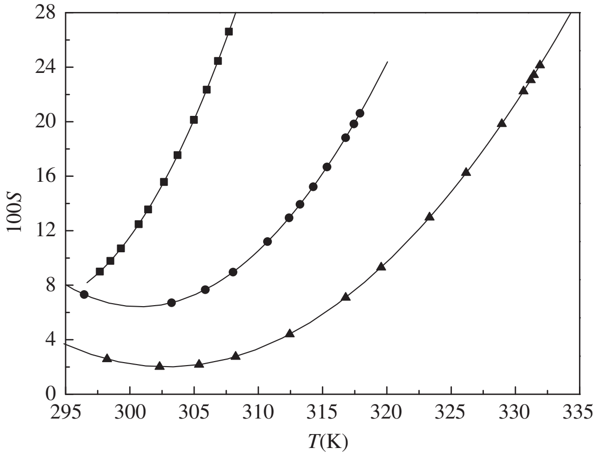

Figure 5. Mass fraction solubilities of [HYDEMIM][BF_4] in ethanol, 1-propanol and 1-butanol. Measured solubilities of [HYDEMIM][BF_4] in: ■, ethanol; ●, 1-propanol; ▲, 1-butanol – calculated according to Equation (1).

4. Results and discussion

The slight difference in the chemical shifts of ^1H for [HYDEMIM][BF_4] maybe due to the different solvents used in the NMR test (methanol-d4) [14]. The other different test conditions, such as temperature, concentration of sample, water content of the sample, etc., could also bring about the variation in the chemical shifts of ^1H for [HYDEMIM][BF_4]. The IR spectra results of [HYDEMIM][BF_4] were in good agreement with the literature [15].

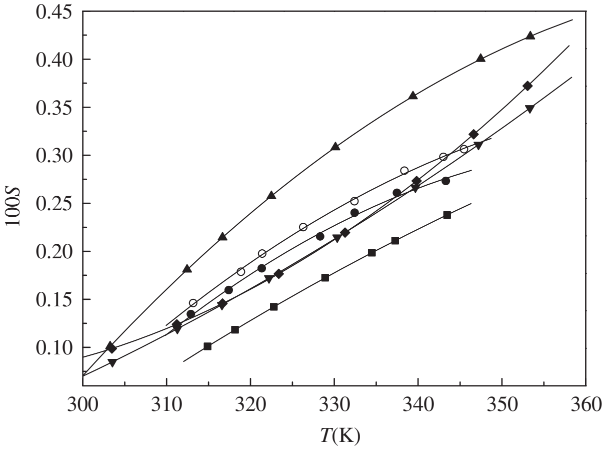

Figure 6. Mass fraction solubilities of [HYDEMIM][BF_4] in benzene, toluene, *o*-xylene, *m*-xylene, *p*-xylene and ethylbenzene. Measured solubilities of [HYDEMIM][BF_4] in: ■, benzene; ●, toluene; ▲, *o*-xylene; ▼, *m*-xylene; ♦, *p*-xylene; ○, ethylbenzene – calculated according to Equation (1).

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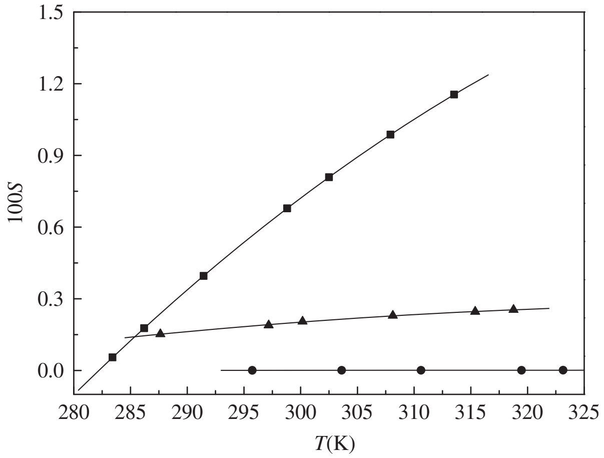

Figure 7. Mass fraction solubilities of [HYDEMIM][BF_4] in dichloromethane, chloroform, carbon tetrachloride. Measured solubilities of [HYDEMIM][BF_4] in: ■, dichloromethane; ●, chloroform; ▲, carbon tetrachloride – calculated according to Equation (1).

Within the temperature range of the measurements, all the binary systems [HYDEMIM][BF_4]–organic solvents exhibited upper critical solution temperatures (UCSTs), as shown in Table 1.

For the alcohol (ethanol, 1-propanol or 1-butanol) investigated, it can be seen from Figures 2 and 5 that the [HYDEMIM][BF_4]–alcohol mutual solubilities decreased with increasing alcohol chain length from 296.45 to 331.91 K, which is consistent with other hydrophilic ILs systems reported in the literature, such as [BMIM][BF_4] with 1-propanol and 1-butanol [11], [OMIM][BF_4] with 1-hexanol and

Table 3. Comparison of the mass fraction solubilities (S) of o-xylene, m-xylene and p-xylene in [HYDEMIM][BF_4] with [BMIM][BF_4] and the separation factor (SF) for the separation of two xylene isomers mixture at $T = 298.15$ K.

	[HYDEMIM][BF_4][a]	[BMIM][BF_4][b]
Solute	$100S$	
o-Xylene	0.0983	0.0194
m-Xylene	0.0820	0.0364
p-Xylene	0.0982	0.0268
Mixture	SF	
o-Xylene(i) + m-xylene(j)	1.199	1.876
o-Xylene(i) + p-xylene(j)	1.001	1.381
p-Xylene(i) + m-xylene(j)	1.198	1.358

[a] Data obtained from this work.

[b] Data obtained from [8].

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1-octanol [12], [$(C_6H_{13}OCH_2)_2$IM][BF_4] with 1-hexanol or 1-octanol or 1-decanol [16], etc. By increasing the alkyl chain of the alcohol, the UCSTs of the system increases because the alcohol becomes more aliphatic, limiting its ability to interact with the ILs through hydrogen bonding, dipolar and Coulombic forces [17].

The binary system [HYDEMIM][BF_4] + benzene derivative (benzene, toluene, o-xylene, m-xylene, p-xylene or ethylbenzene) showed very limited mutual solubility, as shown in Figures 3 and 6. Among the three xylene isomers studied, it was discovered that the [HYDEMIM][BF_4]–xylene isomer mutual solubilities decreased in the order: m-xylene $<$ p-xylene $<$ o-xylene, which may be resulted from the difference in the induced dipole moment related to molecular structure. The similar order is observed for the binary systems [BMIM][BF_4] + xylene isomers [8]. Similarly, [BMIM][BF_4] gave relatively higher separation factors compared to [HYDEMIM][BF_4] for the separation of xylene isomers mixture at 298.15 K, as shown in Table 3.

Partial miscibility between the [HYDEMIM][BF_4] and three chlorinated methanes (CH_2Cl_2, $CHCl_3$, CCl_4) studied was found, as shown in Figures 4 and 7. The ([HYDEMIM][BF_4] + dichloromethane) binary mixture gave relatively higher mutual solubilities, which maybe related to relatively smaller molecular weight of dichloromethane.

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