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Solubilities of 1,1′-(Butane-1,4-diyl)-bis(pyridinium) Dihexafluorophosphate in Acetone + Water from (278.15 to 328.15) K

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Using a laser monitoring technique, the solubilities of 1,1′-(butane-1,4-diyl)-bis(pyridinium) dihexafluorophosphate in acetone (1) + water (2) have been determined experimentally from (278.15 to 328.15) K at atmospheric pressure. The experimental data were correlated with the modified Apelblat equation and the λh model. The calculated results showed good agreement with the experimental data. The solubility of 1,1′-(butane-1,4-diyl)-bis(pyridinium) dihexafluorophosphate in acetone + water is higher than that in water and acetone, respectively, and reaches the maximum value when the mass fraction $w_2 = 0.30$.

Introduction

Ionic liquids (ILs) have been the object of increasing attention due to their unique physicochemical properties such as high thermal stability, high ionic conductivity, high solvating capacity, negligible vapor pressure, and nonflammability that make them ideal solvents for green chemistry and clean synthesis, and ILs have been widely applied to several areas including catalysis, electrochemistry, separation science, biology, and materials for optoelectronic applications.[1−7] The use of ionic liquids as solvents for a broad range of chemical processes is potentially one of the most significant developments of the past decade.[8]

Dicationic ionic liquids have been shown to possess superior physical properties in terms of thermal stability and volatility compared to traditional ionic liquids.[9,10] Consequently, they have been proposed as solvents in high-temperature reactions,[11] novel high-temperature lubricants,[12] and ultrastable separation phases[13,14] and have potential in extractions similar to monocationic ionic liquids and mass spectrometry where ordinary ILs fail.[15] An advantage of dicationic ionic liquids over monocationic ionic liquids is that they provide more opportunities to fine-tune their physical and chemical properties. The synthesis and characterization of a wide variety of dicationic ionic liquids were published while our work was in progress.

To design any process involving ILs on an industrial scale, it is necessary not only to know a range of physical properties including viscosity, density, melting point temperature, electrochemical window, heat capacity, and interfacial tension but also to know other thermodynamic properties including phase equilibria such as vapor−liquid equilibria (VLE), liquid−liquid equilibria (LLE), and solid−liquid equilibria (SLE). An understanding of SLE is of paramount importance for the design of separation processes, especially antisolvent crystallization. There is a pressing need to develop better solvents for separation, especially in the cases of nonideal complexing mixtures. The solubilities of ILs in different solvents have been investigated extensively.[16−21] In this study, the solubilities of solid 1,1′-(butane-1,4-diyl)-bis(pyridinium) dihexafluorophosphate ($[C_4(Py)_2][PF_6]_2$) in an acetone + water binary solvent mixture have been measured from (278.15 to 328.15) K by a laser monitoring observation technique at atmospheric pressure. The experimental data were correlated with the modified Apelblat equation and the λh model.[22−24]

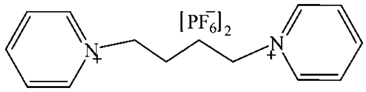

Figure 1. Molecular structure of $[C_4(Py)_2][PF_6]_2$.

Experimental Section

Materials. High-grade 1,1′-(butane-1,4-diyl)-bis(pyridinium) dihexafluorophosphate from Shanghai Chengjie Chemical Co. Ltd. was used directly without further purification, and its purity was determined by high-performance liquid chromatography (type Waters 600E, Waters Co.) to be greater than 99 % in mass fraction and was stored under nitrogen. Analysis for water contamination using the Karl Fischer technique (method Titro-Line KF) for the IL showed that the mass fraction was less than 0.001 %. The melting point temperature (T_m) and the enthalpy of fusion of 1,1′-(butane-1,4-diyl)-bis(pyridinium) dihexafluorophosphate is 552.12 K and 42188.13 $J \cdot mol^{-1}$ measured by differential scanning calorimetry (type Q20, TA Instruments). The molecular structure of $[C_4(Py)_2][PF_6]_2$ is illustrated in Figure 1. High-grade acetone from Tianjin Kemel Chemical Reagent Co., Ltd. was used directly without further purification, and its mass fraction purity was greater than 99 %. The water used in the experiments was double-distilled water.

Apparatus and Procedure. The solubilities were measured by a dynamic method at atmospheric pressure.[22,23] The laser monitoring observation technique[25,26] was used to determine the dissolution temperature of a solid−liquid mixture of known composition. The experiments were carried out in a magnetically stirred, jacketed glass vessel (120 cm^3). A constant temperature (± 0.02 K) was maintained by circulating water through the outer jacket from a super thermostatic water-circulator bath (type HWC-52, Shanghai Cany Precision Instrument Co., Ltd.) at the required temperature. A condenser was connected with the vessel to prevent the solvent from evaporating. A mercury-in-glass thermometer was inserted into the inner chamber of

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Table 1. Mole Fraction Solubilities (*x*) of $[C_4(Py)_2][PF_6]_2$ in (*w*) Water + (1 − *w*) Acetone, Where *w* is the Mass Fraction

T/K	10^2x	10^2x_{Alp}	10^2RD	T/K	10^2x	10^2x_{Alp}	10^2RD
			$w = 0.00$				
278.15	0.0480	0.0480	2.292	308.15	0.0767	0.0769	0.261
283.15	0.0520	0.0525	1.923	313.15	0.0820	0.0822	0.244
288.15	0.0573	0.0571	0.175	318.15	0.0870	0.0875	0.575
293.15	0.0623	0.0619	1.284	323.15	0.0925	0.0929	0.432
298.15	0.0675	0.0668	2.074	328.15	0.0992	0.0983	0.907
303.15	0.0713	0.0718	0.421				
			$w = 0.10$				
278.15	0.0643	0.0654	1.711	308.15	0.1795	0.1816	1.170
283.15	0.0810	0.0804	0.741	313.15	0.2032	0.2057	1.230
288.15	0.0982	0.0973	0.916	318.15	0.2291	0.2304	0.567
293.15	0.1182	0.1161	1.777	323.15	0.2574	0.2555	0.738
298.15	0.1361	0.1365	0.294	328.15	0.2825	0.2805	0.708
303.15	0.1582	0.1584	0.126				
			$w = 0.20$				
278.15	0.0769	0.0772	0.390	308.15	0.2068	0.2065	0.145
283.15	0.0918	0.0938	2.179	313.15	0.2305	0.2341	1.562
288.15	0.1152	0.1125	2.344	318.15	0.2621	0.2630	0.343
293.15	0.1365	0.1332	2.418	323.15	0.2906	0.2929	0.791
298.15	0.1552	0.1558	0.387	328.15	0.3288	0.3234	1.642
303.15	0.1786	0.1803	0.952				
			$w = 0.30$				
278.15	0.1209	0.1218	0.744	308.15	0.2962	0.2993	1.047
283.15	0.1441	0.1448	0.486	313.15	0.3374	0.3380	0.178
288.15	0.1738	0.1703	2.014	318.15	0.3699	0.3790	2.460
293.15	0.1966	0.1986	1.017	323.15	0.4204	0.4222	0.428
298.15	0.2305	0.2295	0.434	328.15	0.4763	0.4675	1.848
303.15	0.2684	0.2631	1.975				
			$w = 0.40$				
278.15	0.0982	0.0982	0.000	308.15	0.2322	0.2286	1.550
283.15	0.1134	0.1123	0.970	313.15	0.2719	0.2651	2.501
288.15	0.1292	0.1289	0.232	318.15	0.3108	0.3080	0.901
293.15	0.1478	0.1482	0.271	323.15	0.3578	0.3582	0.112
298.15	0.1668	0.1709	2.458	328.15	0.4112	0.4172	1.459
303.15	0.1936	0.1974	1.963				
			$w = 0.50$				
278.15	0.0578	0.0589	1.903	308.15	0.1392	0.1429	2.658
283.15	0.0691	0.0681	1.447	313.15	0.1715	0.1660	3.207
288.15	0.0813	0.0789	2.952	318.15	0.1992	0.1928	3.213
293.15	0.0932	0.0914	1.931	323.15	0.2274	0.2239	1.539
298.15	0.1015	0.1061	4.532	328.15	0.2525	0.2601	3.010
303.15	0.1199	0.1231	2.669				
			$w = 0.60$				
278.15	0.0208	0.0208	0.115	308.15	0.0401	0.0404	0.743
283.15	0.0228	0.0230	0.855	313.15	0.0451	0.0447	0.895
288.15	0.0257	0.0255	0.607	318.15	0.0509	0.0518	1.737
293.15	0.0289	0.0285	1.498	323.15	0.0576	0.0566	1.767
298.15	0.0313	0.0318	1.661	328.15	0.0652	0.0657	0.761
303.15	0.0356	0.0357	0.185				
			$w = 0.70$				
278.15	0.0029	0.0029	0.000	308.15	0.0236	0.0251	6.356
283.15	0.0042	0.0046	9.524	313.15	0.0314	0.0320	1.911
288.15	0.0076	0.0069	9.211	318.15	0.0381	0.0397	4.199
293.15	0.0102	0.0100	1.961	323.15	0.0478	0.0480	0.418
298.15	0.0146	0.0141	3.425	328.15	0.0595	0.0566	4.874
303.15	0.0195	0.0191	2.051				
			$w = 0.80$				
278.15	0.0007	0.0007	0.000	308.15	0.0074	0.0077	4.054
283.15	0.0011	0.0011	0.000	313.15	0.0108	0.0109	0.926
288.15	0.0017	0.0017	0.000	318.15	0.0155	0.0151	2.581
293.15	0.0026	0.0025	3.846	323.15	0.0217	0.0208	4.147
298.15	0.0038	0.0037	2.632	328.15	0.0272	0.0281	3.309
303.15	0.0052	0.0054	3.846				
			$w = 0.90$				
278.15	0.0002	0.0002	0.000	308.15	0.0032	0.0032	0.000
283.15	0.0004	0.0004	0.000	313.15	0.0046	0.0045	2.174
288.15	0.0006	0.0006	0.000	318.15	0.0063	0.0063	0.000
293.15	0.0010	0.0010	0.000	323.15	0.0085	0.0085	0.000
298.15	0.0014	0.0015	7.143	328.15	0.0114	0.0113	0.877
303.15	0.0021	0.0022	4.762				
			$w = 1.00$				
278.15	0.0001	0.0001	0.000	308.15	0.0021	0.0022	4.762
283.15	0.0002	0.0002	0.000	313.15	0.0028	0.0029	3.571
288.15	0.0003	0.0003	0.000	318.15	0.0042	0.0041	2.381
293.15	0.0007	0.0007	0.000	323.15	0.0053	0.0052	1.887
298.15	0.0011	0.0011	0.000	328.15	0.0066	0.0065	1.515
303.15	0.0014	0.0015	7.143				

Table 2. Parameters of Equation 3 and Equation 4 for the $[C_4(Py)_2][PF_6]_2$ + Water + Acetone System at Various Contents of Water (w) in the Mixed Solvent

	λh equation		Apelblat equation		
w	λ	h	A	B	C
0.00	0.00243	411241.27	25.50	−2587.49	−4.24
0.10	0.05632	43310.75	193.02	−11236.10	−28.42
0.20	0.06432	37879.06	145.94	−9074.77	−21.41
0.30	0.07946	29216.99	85.39	−6198.41	−12.40
0.40	0.11410	23847.81	−164.14	4853.14	24.83
0.50	0.07539	36652.06	−128.87	3184.60	19.54
0.60	0.00784	266786.17	−156.60	4907.30	23.18
0.70	0.20600	22935.49	552.50	−29849.80	−80.97
0.80	0.80990	7962.33	91.79	−10290.50	−11.84
0.90	0.37691	17370.79	368.10	−23197.70	−52.90
1.00	0.11601	51856.50	891.16	−47052.40	−130.74

the vessels for the measurement of the temperature. The laser monitoring system consists of a laser generator, a photoelectric transformer, and a light intensity display. A laser beam was used to observe the dissolution of the solid−liquid mixture. The light signal transmitted through the vessel was collected by a detector (type FGF-III), which decided the rate of temperature rise and estimated the equilibrium point of acetone + water on the basis of the signal change. In the early stage of the experiment, the laser beam was blocked by the unsolved particles of $C_4(Py)_2$-PF_6 in the solution, so the intensity of the laser beam penetrating the vessel was lower. The intensity increased gradually along with the increase of the amount of $[C_4(Py)_2][PF_6]_2$ dissolved. When the last portion of $[C_4(Py)_2][PF_6]_2$ just disappeared, the intensity of the laser beam penetrating the vessel reached the maximum, and the temperature was recorded.

Solvents for the solubility measurement were prepared by mass using an analytical balance (type XS104, Mettler-Toledo Co.). The balance has a range of measurement up to 120 g, with an uncertainty of ± 0.0001 g. Before the solubility measurement, through the condenser, high-purity nitrogen (99.9995 % by mass, 20 mL·min^{-1}) was fed into the solvent for 2 h to remove the dissolved oxygen. Predetermined amounts of $[C_4(Py)_2][PF_6]_2$ were weighed and transferred into the vessel. The contents of the vessel were heated very slowly at rates less than 1 K·h^{-1} with continuous stirring to approach the solid−liquid equilibrium point. In the processes of solubility measure-

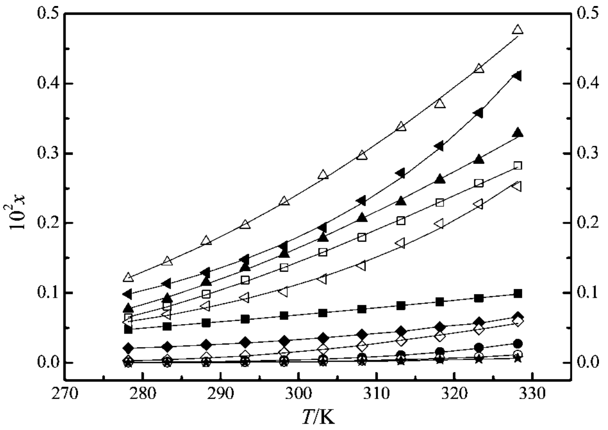

Figure 2. Solubilities of $[C_4(Py)_2][PF_6]_2$ in (*w*) water + (1 − *w*) acetone, where *w* is the mass fraction. ■, *w* = 0.00; □, *w* = 0.10; ▲, *w* = 0.20; △, *w* = 0.30; solid triangle pointing left, *w* = 0.40; open triangle pointing left, *w* = 0.50; ◆, *w* = 0.60; ◇, *w* = 0.70;K; ●, *w* = 0.80; ○, *w* = 0.90; ★, *w* = 1.00; —, calculated values of tie-lines from the Apelblat equation at corresponding temperatures.

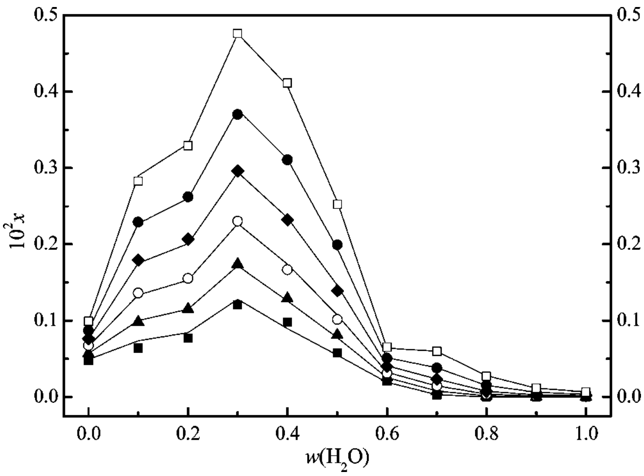

Figure 3. Dependence of the solubilities (x) on the mass fraction (w) of water in the $[C_4(Py)_2][PF_6]_2$ + water + acetone system. ■, $T = 278.15$ K; ▲, $T = 288.15$ K; ○, $T = 298.15$ K; ◆, $T = 308.15$ K; ●, $T = 318.15$ K; □, $T = 328.15$ K; —, calculated values of tie-lines from the λh model at corresponding temperature.

ment, some of the solubility experiments were conducted at least three times to check the reproducibility, and the mean values were considered as the measured results. The reproducibility of the measurements was 0.1 K, which corresponds to a relative error in composition smaller than ± 1 %.[27] In this work, the uncertainty for solubility measurement is estimated on the basis of the principle of the error propagation to be 2.0 % at the 95 % confidence level.

Results and Discussion

To our knowledge, this is the first time the solubilities of $[C_4(Py)_2][PF_6]_2$ ILs have been published. The measured mole fraction solubilities (x) of $[C_4(Py)_2][PF_6]_2$ in acetone (1) + water (2) at different temperatures (T) are presented in Table 1. The mass fraction (w) of water in the solvents was 0, 0.10, 0.20, 0.30, 0.40, 0.50, 0.60, 0.70, 0.80, 0.90, and 1.00. The experimental points and calculated values were shown in Table 1, where T is the absolute temperature and x and x_{alp} are the experimental and calculated values of the modified Apelblat equation.[24,28] RD is the relative deviation which is defined as follows

$$\mathrm{RD} = \frac{|x_{\mathrm{calcd}} - x|}{x} \tag{1}$$

The average relative deviation (ARD) is given as follows

$$\mathrm{ARD} = \frac{\sum_{1}^{n} \mathrm{RD}}{n} \tag{2}$$

where n is the number of experimental values.

The relationship between mole fraction of the solubility and temperature is described by the λh model and the modified Apelblat equation. The λh model which is a semiempirical equation is shown as follows

$$\ln\left[1 + \frac{\lambda(1-x)}{x}\right] = \lambda h\left[\frac{1}{(T/\mathrm{K})} - \frac{1}{(T_{\mathrm{m}}/\mathrm{K})}\right] \tag{3}$$

where λ and h are the model parameters determined by the experimental data and are listed in Table 2; x is the mole fraction of the solubility of IL at the system temperature T; and T_{m} is the normal melting temperature.

The modified Apelblat equation is shown as follows

$$\ln x = A + \frac{B}{T/\mathrm{K}} + C \ln(T/\mathrm{K}) \tag{4}$$

where x is the mole fraction solubility of IL; T is the absolute temperature; and A, B, and C are empirical constants. The values of A, B, and C obtained from the experimental solubility data in the systems are listed in Table 2.

From Table 1 and Figure 2, the calculated solubilities are in good agreement with the experimental data, which indicate that the modified Apelblat equation can be used to correlate the solubility data of $[C_4(Py)_2][PF_6]_2$ in acetone + water. The ARD values for the Apelblat equation and the λh model were 0.016 and 0.025, respectively, which showed that the Apelblat equation was more accurate than the λh equation for this system. The experimental solubility and correlation equation in this work can be used as essential data and models to serve the synthesis and purification process of $[C_4(Py)_2][PF_6]_2$.

By using the data shown in Table 1, the dependence of the solubilities x calculated from the λh model on the mass fraction w of water in solvent for the $[C_4(Py)_2][PF_6]_2$ + acetone + water system were given in Figure 3. It showed the relations between the solubility and the composition of the mixed solvent at fixed temperatures. From the results shown in Table 1 and Figure 3, it can be seen that the solubility of $[C_4(Py)_2][PF_6]_2$ in the acetone + water mixture is higher than that in water and acetone, respectively. It also can be seen from Figure 3 that solubility of $[C_4(Py)_2][PF_6]_2$ in the acetone + water system has a maximum value at $w = 0.30$. According to Scatchard–Hildebrand's theory,[29] the solubility of a solid is a maximum in that solvent whose solubility parameter is the same as that of the (liquid) solute; therefore, a plot of solubility versus solvent composition should go through a maximum. Therefore, the solubility of the solute in the solvent is the largest when the solubility parameters (δ) of the solute and the solvent are the same. For the binary solvent system of A and B, it is possible that the solubility got to be the maximum when the solubility parameter met the following relationship: $\delta_{\mathrm{A}} < \delta_1 < \delta_{\mathrm{B}}$. The values of the solubility parameter of $[C_4(Py)_2][PF_6]_2$, acetone, and water are 25.19 $\mathrm{J}^{1/2}\cdot\mathrm{cm}^{-3/2}$ (measured by the inverse gas chromatography method[30]), 20.05 $\mathrm{J}^{1/2}\cdot\mathrm{cm}^{-3/2}$, and 47.90 $\mathrm{J}^{1/2}\cdot\mathrm{cm}^{-3/2}$, respectively, so the solubility of $[C_4(Py)_2][PF_6]_2$ may get to be the maximum in the binary solvent system of acetone and water, which may be used to serve the purification process of $[C_4(Py)_2][PF_6]_2$.

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