Phase Diagram Data for Several Salt + Salt Aqueous Biphasic Systems at 298.15 K

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Phase diagrams corresponding to aqueous biphasic systems of salt (the organic ionic liquid of salts [C_4mim]Cl, [C_6mim]Cl, and [C_8mim]Cl) + salt (K_3PO_4, K_2CO_3) + water were determined at 298.15 K. The binodal curve was fitted to the Merchuk equation. Tie lines assigned from mass phase ratios according to the lever arm rule were satisfactorily described using the Othmer−Tobias and Bancroft equations.

Introduction

Aqueous biphasic systems (ABS) are formed when two mutually incompatible polymers/polymer, polymer/salt, or salt/salt are dissolved in water above a certain critical concentration. These systems comprise mass fraction of water of (80 to 90) % in equilibrium phases, each phase containing mainly one of the compounds and a small amount of the other. ABS have been widely used in the separation, concentration, and fractionation of biological solutes and particles such as cells and proteins.[1,2] ABS have also been studied for applications including the selective distribution and separation of metal ion species,[3] small organic molecules,[4,5] nano- and micro-solid particulates,[6,7] and as green reaction media.[8−10] In recent years, ionic liquids (ILs) have been gaining great exposure due to their potential use as green solvents and as possible replacements for traditional volatile organic compounds (VOCs). A new type of ABS consisting of ILs and salt was reported for recycle or concentration hydrophilic ILs from aqueous solution. Rogers et al.[11] first reported that 1-butyl-3-methylimidazolium chloride ([C_4mim]Cl) + K_3PO_4 + water can be used to form an ABS. Li and co-workers[12,13] studied the extraction of biochemical compounds and drugs using [C_4mim]Cl + K_2HPO_4 + water ABS. Akama et al.[14,15] used a tetrabutylammonium bromide + $(NH_4)_2SO_4$ + water ABS to separate metal ions. Bridges et al.[16] described linear solvent free-energy relationships and Gibbs free energy of methylene transfer in salt + salt + water ABS. However, data for the phase diagram of the salt + salt + water systems are not available at the present.

In our previous work, ILs were concentrated from aqueous solutions by aqueous biphasic systems.[17] The goal of this study is to obtain salt (imidazolium ILs of [C_4mim]Cl, [C_6mim]Cl, and [C_8mim]Cl) + salt (K_3PO_4, K_2CO_3) + water ABS phase diagrams at 298.15 K.

Experimental Section

Materials. K_3PO_4 and K_2CO_3 were analytical grade reagents (Beijing Chemical, Beijing, China), and all the solutions were prepared from deionized water. The ILs used in this study were prepared by the following procedures:

(1) 1-Octyl-3-methylimidazolium chloride ([C_8mim]Cl). Freshly distilled 1-chlorooctane (5 L) and *N*-methylimidazole (2.32 L) were added in a 20 L reactor (Shensheng, Shanghai, China) with a mechanical stirrer and a condenser. The solution was stirred for 70 h at 80 °C. The [C_8mim]Cl was easily separated from the small amount of unreacted compounds by separating funnels and washed three times with ethyl acetate (3 × 3 L). The ethyl acetate was finally removed using a vacuum line. Thus, the pure [C_8mim]Cl was obtained (5.7 L, yield 84.2 %). The purity of the compound was verified through ^{1}H NMR and ^{13}C NMR (Bruker AV 400 NMR). The water content was determined by a Karl Fischer titration using a WA-1C moisture meter (Jiangfen, Jiangsu, China).

(2) 1-Hexyl-3-methylimidazolium chloride ([C_6mim]Cl). The same procedure was used, except that 1-chlorohexane was substituted for 1-chlorooctane.

(3) 1-Butyl-3-methylimidazolium chloride ([C_4mim]Cl). The same procedure was used, except that 1-chlorobutane was substituted for 1-chlorooctane.

Methods. The binodal curve was determined by the cloud point method at room temperature.[1] From the stock, an IL solution of known concentration was taken into the vessel. A salt solution of known mass fraction was then added dropwise to the vessel until the mixture became turbid or cloudy. The composition of this mixture was noted. Then, water was added dropwise to the vessel to get a clear one-phase system, and more salt solution was added again to form the ABS, and the above procedure was repeated and so on. The binodal curve was fitted by least-squares regression to an empirical relationship developed by Merchuk[18] and shown as eq 1:

$$Y_A = a \exp(bX_A^{0.5} - cX_A{}^3) \tag{1}$$

where Y_A and X_A are the concentrations of IL and salt. The constants *a*, *b*, and *c* were obtained by least-squares regression. The tie line was determined by application of the lever arm rule to the relationship between the mass phase composition and the overall system composition.[18] The mathematical methods are shown in eqs 2 and 3:

$$Y_T = (Y_M/R) - ((1 - R)/R)Y_B \tag{2}$$

$$X_T = (X_M/R) - ((1 - R)/R)X_B \tag{3}$$

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Table 1. Binodal Data for Salt (1) + Salt (2) + Water (3) ABS at 298.15 K

$[C_4mim]Cl + K_3PO_4$		$[C_4mim]Cl + K_2CO_3$		$[C_6mim]Cl + K_3PO_4$		$[C_6mim]Cl + K_2CO_3$		$[C_8mim]Cl + K_3PO_4$		$[C_8mim]Cl + K_2CO_3$	
$100\, w_2$	$100\, w_1$	$100\, w_2$	$100\, w_1$	$100\, w_2$	$100\, w_1$	$100\, w_2$	$100\, w_1$	$100\, w_2$	$100\, w_1$	$100\, w_2$	$100\, w_1$
15.48	18.73	13.69	25.48	7.41	41.94	8.41	44.51	13.55	28.83	9.22	40.9
16.68	16.68	16.62	20.89	9.44	35.63	10.44	36.57	14.66	25.24	11.56	34.23
17.84	15.38	18.95	17.67	13.22	25.27	13.55	29.66	16.87	21.63	13.34	29.27
19.12	13.72	20.73	15.19	14.02	22.60	14.52	26.25	18.37	18.84	15.51	24.74
20.37	12.19	21.98	13.34	15.84	19.47	16.91	21.97	20.04	16.03	17.09	21.53
21.39	10.99	23.77	11.19	17.20	17.05	18.50	19.09	21.26	14.39	18.28	19.04
22.36	9.97	25.18	9.69	18.04	15.17	19.53	16.86	22.15	12.95	19.61	16.97
22.65	9.57	26.49	8.34	19.24	13.53	21.16	14.57	23.33	11.43	20.16	15.98
23.76	8.53	27.64	7.25	20.41	11.89	22.31	12.77	24.38	10.12	21.29	14.20
24.41	7.84	28.65	6.35	21.21	10.98	23.35	11.32	25.66	8.75	22.23	12.80
24.85	7.46	29.52	5.61	21.60	10.23	23.77	10.42	26.52	7.78	22.94	11.69
25.49	6.81	30.38	4.95	21.96	9.57	24.54	9.45	27.16	7.07	23.5	10.72
25.98	6.32	31.74	4.08	22.89	8.63	25.30	8.55	27.87	6.39	23.84	10.29
26.37	6.02	33.28	3.19	23.99	7.80	26.04	7.76	28.73	5.70	24.52	9.4
27.03	5.51			24.50	7.34	26.80	7.03	29.36	5.16	24.93	8.77
27.33	5.29			24.84	6.90	27.14	6.61	29.90	4.72	25.13	8.48
27.87	4.87			25.12	6.52	27.70	6.11	30.74	4.16	25.62	7.88
28.08	4.69			26.15	5.55	28.72	5.23	31.65	3.61	26.07	7.36
28.70	4.27			26.34	5.25	29.63	4.51	32.56	3.13	26.94	6.51
29.34	3.88			26.84	4.71	30.44	3.96	34.31	2.31	27.453	6.05
29.70	3.62			27.66	4.12	31.18	3.45	35.53	1.78	28.13	5.51
29.99	3.46			28.07	3.78	32.06	2.96			30.42	3.76
30.65	3.11					32.77	2.58			30.76	3.56
30.96	2.95					33.67	2.12				
31.88	2.51										

Table 2. Parameters of Equation 1 for Salt (1) + Salt (2) + Water (3) ABS

system	a	b	c	R	σ^a
1 $[C_4mim]Cl/K_3PO_4$	77.19	−0.3132	5.123×10^{-5}	0.9998	0.065
2 $[C_4mim]Cl/K_2CO_3$	83.80	−0.2914	4.347×10^{-5}	0.9999	0.069
3 $[C_6mim]Cl/K_3PO_4$	152.47	−0.4635	5.385×10^{-5}	0.9993	0.26
4 $[C_6mim]Cl/K_2CO_3$	149.52	−0.4118	4.802×10^{-5}	0.9993	0.29
5 $[C_8mim]Cl/K_3PO_4$	144.80	−0.4141	4.275×10^{-5}	0.9994	0.19
6 $[C_8mim]Cl/K_2CO_3$	132.49	−0.3741	5.399×10^{-5}	0.9997	0.16

where M, T, and B represent the mixture, the top phase, and the bottom phase, respectively; Y and X are the concentration of IL and salt; and R is the following measured ratio:

$$R = (\text{weight of the top phase})/(\text{weight of the mixture}) \quad (4)$$

This is a system of four equations with four unknowns that can easily be solved, and the equation for the tie lines can be obtained:

$$Y = Y_0 + S \quad (5)$$

where the slope (S) is given by

$$S = (Y_T - Y_B)/(X_T - X_B) \quad (6)$$

and Y_0 is the point corresponding to $X = 0$. The tie line lengths (TLL) for the different compositions were calculated by the equivalent expression:

$$\text{TLL} = [(Y_T - Y_B)^2 + (X_T - X_B)^2]^{1/2} \quad (7)$$

Results and Discussion

The binodal data for salt + salt + water systems are given in Table 1. The binodal curves for the various salt + salt systems are represented in Figure 1. The data were fitted according to the empirical relationship of Merchuk, and the parameters for this equation as determined by least-squares regression of the cloud point data are shown in Table 2. Huddleston et al. have studied phase diagram data for several PEG + salt + water ABS at 25 °C using this approach and obtained good results.[19] Literature results confirmed that the method can satisfactorily correlate binodal curves and determined tie lines in $[C_4mim]Cl + K_3PO_4$ ABS.[11] The coefficients of eq 1 along with the corresponding correlation coefficient values (R) and standard deviations (σ^a) are given in Table 2, it indicated that the empirical equation (eq 1) gave a rather good fit to the data for descriptive purposes. Tie lines for these cloud point curves were fitted by a least-squares method based on the lever arm rule described previously. Numerical data for these tie lines are given in Table 3. The average slopes of the tie lines are shown in Table 3. As may be seen from these data, the slopes of the tie lines were approximately constant at short tie line lengths but showed significant change as tie line length increased. This was probably due to the fact that the salt phase was virtually free of IL at long tie line lengths.[19] These data are also in agreement with that for $[C_4mim]Cl + K_3PO_4$ ABS initially reported by Gutowski et al.[11]

We can generalize the formation of ABS of this type by considering the use of other water structuring salts to salt-out the IL ions from aqueous solution.[9] Figure 1 illustrates the binodal curves for $[C_4mim]Cl$, $[C_6mim]Cl$, and $[C_8mim]Cl$ with K_3PO_4 and K_2CO_3 ABS. It shows that the binodal curves for

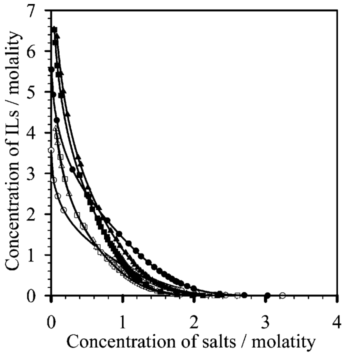

Figure 1. Phase diagram for $[C_nmim]Cl$ (1) + salts (2) + water (3) ABS at 298.15 K: ○, $[C_4mim]Cl + K_3PO_4$ + water; △, $[C_6mim]Cl + K_3PO_4$ + water; □, $[C_8mim]Cl + K_3PO_4$ + water; ●, $[C_4mim]Cl + K_2CO_3$ + water; ▲, $[C_6mim]Cl + K_2CO_3$ + water; ■, $[C_8mim]Cl + K_2CO_3$ + water.

Table 3. Tie Line Composition of Salt (1) + Salt (2) + Water (3) Assigned by the Lever Arm Rule

[C$_4$mimCl] (1) + K$_3$PO$_4$ (2) + Water (3)

	tie 1	tie 2	tie 3	tie 4
100 w_1(mixture)	15.22	16.63	18.12	22.16
100 w_2(mixture)	25.04	27.89	30.08	39.99
100 w_1(top)	44.49	51.76	60.16	75.63
100 w_1(bottom)	1.19	0.40	0.19	0.0007
100 w_2(top)	3.08	1.63	0.63	0.0042
100 w_2(bottom)	35.57	40.02	42.64	56.56
R	0.324	0.316	0.299	0.293
TLL	54.13	64.12	73.22	94.44
S	−1.33	−1.34	−1.43	−1.34

[C$_4$mimCl] (1) + K$_2$CO$_3$ (2) + Water (3)

	tie 1	tie 2	tie 3	tie 4	tie5
100 w_1(mixture)	15.08	15.99	16.98	18.14	18.94
100 w_2(mixture)	23.01	24.86	30.12	34.84	39.85
100 w_1(top)	33.61	42.75	59.47	68.13	76.63
100 w_1(bottom)	4.66	2.39	0.62	0.12	0.016
100 w_2(top)	9.13	5.24	1.38	0.50	0.094
100 w_2(bottom)	30.82	34.83	41.18	47.22	52.89
R	0.360	0.337	0.278	0.265	0.247
TLL	36.17	50.04	71.04	82.51	93.04
S	−1.33	−1.36	−1.47	1.45	−1.45

[C$_6$mimCl] (1) + K$_2$CO$_3$ (2) + Water (3)

	tie 1	tie 2	tie 3	tie 4
100 w_1(mixture)	15.18	17.24	19.00	21.09
100 w_2(mixture)	25.33	29.98	34.84	39.87
100 w_1(top)	36.47	60.72	72.16	83.03
100 w_1(bottom)	0.69	0.25	0.031	0.0018
100 w_2(top)	9.01	3.92	2.60	1.72
100 w_2(bottom)	36.44	40.17	46.35	52.86
R	0.405	0.281	0.263	0.254
TLL	45.08	70.50	84.36	97.51
S	−1.30	−1.67	−1.65	−1.62

[C$_6$mimCl] (1) + K$_2$CO$_3$ (2) + Water (3)

	tie 1	tie 2	tie 3	tie 4	tie5
100 w_1(mixture)	15.12	16.04	17.07	18.10	19.81
100 w_2(mixture)	25.13	27.92	30.08	32.01	37.79
100 w_1(top)	46.89	61.44	69.26	75.44	87.91
100 w_1(bottom)	3.19	1.55	0.77	0.39	0.039
100 w_2(top)	7.64	4.62	3.48	2.75	1.66
100 w_2(bottom)	31.70	35.36	38.39	41.05	48.28
R	0.273	0.242	0.238	0.236	0.225
TLL	49.89	67.32	76.87	84.26	99.47
S	−1.82	−1.95	−1.96	−1.96	−1.88

[C$_8$mimCl] (1) + K$_3$PO$_4$ (2) + Water (3)

	tie 1	tie 2	tie 3	tie 4
100 w_1(mixture)	16.13	17.95	19.52	20.46
100 w_2(mixture)	25.49	30.03	35.18	38.71
100 w_1(top)	52.93	67.48	79.56	86.95
100 w_1(bottom)	3.20	0.82	0.14	0.035
100 w_2(top)	5.81	3.38	2.09	1.52
100 w_2(bottom)	32.40	39.25	45.86	50.16
R	0.260	0.257	0.244	0.235
TLL	56.39	75.70	90.68	99.60
S	−1.87	−1.85	−1.81	−1.79

[C$_8$mimCl] (1) + K$_2$CO$_3$ (2) + Water (3)

	tie 1	tie 2	tie 3	tie 4	tie 5
100 w_1(mixture)	18.31	19.19	19.64	20.10	21.01
100 w_2(mixture)	27.11	31.00	34.69	37.88	41.43
100 w_1(top)	67.91	74.91	78.06	85.84	90.17
100 w_1(bottom)	1.33	0.32	0.062	0.017	0.002
100 w_2(top)	3.18	2.32	2.00	1.35	1.06
100 w_2(bottom)	35.30	40.71	45.65	49.04	53.69
R	0.255	0.253	0.251	0.234	0.233
TLL	73.92	83.89	89.38	98.18	104.40
S	−2.07	−1.94	−1.79	−1.79	−1.71

the same IL with different salts ABS, and K$_3$PO$_4$ results in the binodal curve closer than K$_2$CO$_3$ to the origin, meaning less salt is needed to form an ABS. It indicated that the salting-out ability of the anions follows the ordering PO$_4^{3-}$ > CO$_3^{2-}$. This ordering directly follows the anions calculated ΔG_{hyd} values (all ΔG_{hyd} date from ref 3, PO$_4^{3-}$, −2835 kJ·mol^{-1} > CO$_3^{2-}$, −1300 kJ·mol^{-1}).

The correlating equations proposed by Othmer−Tobias (eq 8) and Bancroft (eq 9) have been used to correlate the tie line compositions:

$$\left(\frac{1-W_1^{\mathrm{t}}}{W_1^{\mathrm{t}}}\right)=K\left(\frac{1-W_2^{\mathrm{b}}}{W_2^{\mathrm{b}}}\right)^n \tag{8}$$

$$\left(\frac{W_3^{\mathrm{b}}}{W_2^{\mathrm{b}}}\right)=K_1\left(\frac{W_3^{\mathrm{t}}}{W_1^{\mathrm{t}}}\right)^r \tag{9}$$

where W_1^{t} is the mass fraction of ILs in the top phase; W_2^{b} is the mass fraction of salt in the bottom phase; W_3^{b} and W_3^{t} are the mass fractions of water in the bottom and top phases, respectively; and K, n, K_1, and r are the fit parameters. Equations 8 and 9 are linearized by taking the logarithm on both sides of the equations to determine the fit parameters. The values of the parameters are given in Table 4.

Recently, eqs 8 and 9 have been successfully used for the correlation of the tie line compositions of the PEG + sodium citrate + water and PEG + potassium citrate + water systems.[20,21] A linear dependency of the plots $\log[(1 - W_1^{\mathrm{t}})/W_1^{\mathrm{t}}]$ against $\log[(1 - W_2^{\mathrm{b}})/W_2^{\mathrm{b}}]$ and $\log(W_3^{\mathrm{b}}/W_2^{\mathrm{b}})$ against $\log(W_3^{\mathrm{t}}/W_1^{\mathrm{t}})$ indicated an acceptable consistency of the results. The corresponding correlation coefficient values (R) are also given in Table 4. Furthermore, on the basis of the standard deviations (σ^{a}) given in Table 4, eqs 8 and 9 can be satisfactorily used to correlate the tie line data of the investigated systems.

In the new salt + salt ABS, there are four different ions and speciation of the two different cations and anions. Upon phase separation these ions partition to one of the two phases while maintaining electroneutrality. The most chaotropic ion of each type partitions to the upper phase. Increased kosmotropic salts concentrations enhance phase separation. As the TLL increases, the bottom phase becomes increasingly kosmotropic. The phase is becoming more structured, and thus forcing the chaotropic ion to the upper phase with concurrent transfer of chloride anions. The increased water structuring of the kosmotropic phase decreases the difference in the distribution ration between [C$_n$mim]$^+$ and Cl$^-$.[16]

Conclusions

For salt ([C$_4$mim]Cl, [C$_6$mim]Cl, and [C$_8$mim]Cl) + salt (K$_3$PO$_4$, K$_2$CO$_3$) + water ABS, binodal data and tie line data were measured at 298.15 K. The binodal data was fit with the Merchuk equation relating the concentration of the ILs and salt. The tie line data for these systems were correlated satisfactorily by the Othmer−Tobias and Bancroft equations.

Table 4. Values of the Parameters of Equations 8 and 9

systems	K	n	R	σ^{a}	K_1	r	R	σ^{a}
1 [C$_4$mim]Cl/K$_3$PO$_4$	0.4715	1.5649	0.9898	0.044	1.5691	0.6008	0.9866	0.030
2 [C$_4$mim]Cl/K$_2$CO$_3$	0.3695	2.0253	0.9965	0.032	1.5443	0.4329	0.9909	0.022
3 [C$_6$mim]Cl/K$_3$PO$_4$	0.2614	2.9862	0.9696	0.11	1.3897	0.2613	0.9375	0.046
4 [C$_6$mim]Cl/K$_2$CO$_3$	0.1104	2.9714	0.9988	0.019	1.9301	0.3051	0.9957	0.011
5 [C$_8$mim]Cl/K$_3$PO$_4$	0.1630	2.3668	0.9964	0.035	2.0153	0.4012	0.9963	0.014
6 [C$_8$mim]Cl/K$_2$CO$_3$	0.1601	1.9375	0.9737	0.067	2.3747	0.4806	0.9686	0.035

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