Liquid−Liquid Equilibria of Aqueous Biphasic Systems Composed of 1-Butyl-3-methyl Imidazolium Tetrafluoroborate + Sucrose/Maltose + Water

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Phase behaviors of aqueous biphasic systems (ABSs) composed of 1-butyl-3-methyl imidazolium tetrafluoroborate ([bmim]BF_4) + sucrose/maltose + water were reported in this work. Phase diagrams, including binodal curves, tie-lines, and the slope of tie-line (STL) were obtained at temperatures of T = (278.15, 288.15, 298.15, and 308.15) K. The binodal curve data were correlated using an empirical equation, and the tie-line data were correlated according to Othmer−Tobias and Bancroft equations. Good agreements with the experimental data were obtained, and results showed that these equations can be satisfactorily used to correlate the experimental data of the investigated systems at the given temperatures.

Introduction

Liquid−liquid extraction is an effective separation method and also a promising alternative in analytical science and in chemical industry. However, the traditional liquid−liquid extraction methods are mostly involved in organic solvents, which are usually volatile, toxic, and flammable. Therefore, the more effective and cleaner extraction methods are highly desired for the development of new liquid−liquid extraction processes.

Aqueous biphasic systems (ABSs) and ionic liquids (ILs) are well-known green solvents and have great promise to replace conventional volatile organic compounds (VOCs) involved in extraction systems. ILs and ABSs have been combined together for the first time in 2003.[1] Compared with the polymer−polymer/salt ABSs, IL-based ABSs avoid the disadvantages such as high cost, high viscosity, a long time for phase splitting, and the high concentration of salt. Considering the unique properties of ILs,[2–5] for example, extremely low vapor pressure, nonflammablility, excellent solvent power for organic and inorganic compounds, easy modification structurally to elicit desired physical properties, and excellent performance in the application of biocatalysis, electrochemistry, separation, reaction, and so forth, ABSs based on ILs have great potential applications in chemical engineering and biological engineering.

The mostly reported IL-based ABSs are usually involved in the hydrophilic IL, 1-butyl-3-methyl imidazolium tetrafluoroborate ([bmim]BF_4), such as in [bmim]BF_4 + salt + water,[1,6–9] [bmim]BF_4 + carbohydrate + water,[10–14] and [bmim]BF_4 + amino acid + water systems.[15] These systems have been widely used in extraction, such as that of alcohols,[1,15] antibiotics,[7] phenol,[14] amino acids,[8] and enzymes,[8] and good results were obtained, which shows great potential in purification and separation application. In addition, there is a lot of work[16–18] on the IL and carbohydrates binary system, which provides important information in the investigation of the ternary IL-based ABSs. In this work, we reported the phase behaviors of [bmim]BF_4 + sucrose/maltose + water systems at different temperatures of T = (278.15, 288.15, 298.15, and 308.15) K and 1 atm.

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Experimental Section

Materials. [bmim]BF_4 was prepared according to the reported procedures[19] and was verified by ^{1}H NMR. The synthesized IL was dried under vacuum at 353 K for 48 h. The water content of IL was less than 200 ppm measured by Karl Fischer titration (Metrohm KF 787). Residual chloride in IL was $2 \cdot 10^{-3}$ $mol \cdot L^{-1}$, which was determined by using the chloride-selective electrode.[20] Sucrose and maltose were analytical reagents and were purchased from Beijing Chemistry Reagent Company. Sucrose and maltose were dried under vacuum at 353 K for 48 h and were used without other purification. Water was twice distilled.

Experimental Methods. The experimental apparatus employed was essentially similar to literature.[21] The binodal curves were determined by the cloud-point method at T = (278.15, 288.15, 298.15, and 308.15) K and 1 atm. The uncertainty in the temperature was ± 0.05 K.

The tie-line experimental was prepared by mixing 5 g of sugar solution and 5 g of IL solution in the sealed glass vessel. The mixture was vigorously stirred for 3 h and then was set in the thermostat with a desired temperature for 24 h to separate thoroughly. After separation of the two phases, samples of both phases were collected with a long pinhead syringe and were analyzed.

The concentrations of the IL in the top and bottom phases were determined by high-performance liquid chromatography (HPLC). The analysis experiments were performed on a Agilent 1100 series HPLC with a cation exchange column[22] (Zorbax Scx 250*4.6 mm, I.D. 5 μm, Agilent) and a spectromonitor UV−vis detector (Agilent G1314A). The mobile phase used for the reversed phase separations was an acetonitrile−water (0.50 volume fraction) mixture with KH_2PO_4 ($2.72\ g \cdot L^{-1}$), and flow rate was $1.0\ mL \cdot min^{-1}$. The concentration of water was determined by KF 787 (Metrohm). The concentration of sugar was obtained by the difference. All experiments were determined in duplicate, and the accuracy is about ± 0.001.

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Table 1. Binodal Curve Data for [Bmim]BF_4 (1) + Sucrose/Maltose (2) + H_2O (3) Systems at Different Temperatures

T = 278.15 K		T = 288.15 K		T = 298.15 K		T = 308.15 K	
100 w_1	100 w_2	100 w_1	100 w_2	100 w_1	100 w_2	100 w_1	100 w_2
[C_4mim]BF_4 (1) + Sucrose (2) + H_2O (3)							
2.38	64.00	2.83	59.02	3.85	57.43	5.15	53.88
2.89	58.72	3.69	54.38	5.01	52.71	6.38	49.31
3.27	55.79	3.97	52.55	6.27	48.66	7.17	44.83
3.95	53.70	5.41	48.03	6.55	45.13	8.47	42.07
4.06	51.29	5.73	44.11	8.16	41.58	9.17	39.66
5.18	43.12	6.36	41.65	8.93	37.99	11.24	36.47
5.36	42.25	7.53	38.98	10.01	36.00	12.50	33.70
6.09	38.75	10.71	31.70	11.60	32.94	15.36	30.49
6.21	36.18	12.00	28.20	12.70	29.33	17.00	27.27
6.90	34.04	12.04	27.80	15.24	24.26	21.07	22.42
7.26	32.55	13.20	23.60	17.04	21.59	22.95	21.20
7.60	30.8	14.61	21.28	17.40	21.30	24.76	19.71
7.99	26.40	16.14	18.78	20.24	18.33	26.57	18.59
9.18	23.79	19.40	14.48	22.26	16.01	26.89	18.11
10.10	21.20	21.63	12.13	23.76	14.92	29.94	15.85
11.04	17.84	26.56	8.67	26.58	13.35	35.41	12.83
15.03	11.73	31.74	5.97	28.33	12.03	42.10	10.04
18.45	8.87	35.76	5.25	31.63	10.76	45.09	8.98
21.12	6.79	38.97	4.50	35.27	9.78	48.33	7.59
23.68	5.69	44.96	3.58	38.64	8.50	54.19	5.89
31.80	3.96	53.11	1.73	44.10	6.89	59.57	4.44
43.34	2.32	60.69	1.53	46.66	6.32	63.46	3.31
51.69	1.91	65.11	1.20	48.57	5.69	65.44	2.90
61.40	1.27	65.97	1.15	52.07	5.00	65.80	2.87
67.00	0.90	69.55	0.99	52.60	4.83	67.45	2.57
72.01	0.58	73.30	0.87	54.29	4.44	70.13	2.19
76.20	0.55	83.44	0.56	57.38	3.64	71.90	1.83
78.93	0.50	85.02	0.55	61.08	2.88	73.27	1.71
82.26	0.33	91.40	0.50	63.46	2.61	75.91	1.51
85.90	0.32			65.44	2.40	80.80	1.38
90.06	0.20			67.45	2.17	90.80	1.16
94.50	0.20			70.13	1.79		
				73.10	1.51		
				73.27	1.43		
				80.20	1.38		
				86.80	1.16		
[C_4mim]BF_4 (1) + Maltose (2) + H_2O (3)							
8.20	35.98	10.57	32.70	6.80	46.04	7.82	43.26
8.70	34.14	11.00	31.12	12.61	30.15	9.84	39.11
9.75	27.93	11.93	27.63	16.49	23.65	12.92	33.07
10.07	25.08	12.95	23.61	19.62	19.37	15.82	27.41
11.62	20.41	13.86	21.58	26.96	12.29	19.85	22.76
13.54	17.69	15.41	17.90	32.87	9.57	23.50	18.72
16.80	11.95	19.08	13.52	38.75	7.71	27.22	16.15
19.15	7.98	21.51	11.15	44.52	6.06	35.87	12.07
21.30	6.04	25.78	8.16	49.50	4.58	40.76	10.13
24.39	3.96	29.19	7.10	53.25	3.82	43.04	9.09
27.24	2.80	31.48	5.84	60.17	2.53	45.58	8.22
30.27	1.88	33.71	5.36	67.98	1.49	48.03	7.07
36.57	0.72	40.34	3.87			52.72	5.53
39.42	0.24	48.13	2.73			60.37	3.65
49.16	0.04	51.78	2.42			63.73	3.28
		60.56	1.64			72.99	1.50
		64.18	1.25				
		65.94	1.18				
		70.20	0.90				
		70.80	0.80				
		75.43	0.73				

Results and Discussion

Binodal Curves. The binodal curves of [bmim]BF_4 (1) + sucrose/maltose (2) + H_2O (3) systems were measured at different temperatures of T = (278.15, 288.15, 298.15, and 308.15) K, and the results are shown in Table 1 and Figures 1 and 2. From Figures 1 and 2, it can be seen that the two-phase areas are expanded with decreasing temperature. This phenomenon agrees with that observed in aqueous two-polymer systems,[23] while it is contrary to that observed in single polymer systems.[24] The two-phase areas of the systems are almost similar to each other at the same conditions. This may be due to the interactions between sucrose/maltose and water. Maltose and sucrose have the tendency to form intramolecular hydrogen bonds,[25] which holds back interactions between sugar and water. The published binodal curves of the [bmim]BF_4 + sucrose + water system[11] at T = 308.15 K are also shown in Figure 1, which is consistent with this work.

The binodal curve data for [bmim]BF_4 (1) + sucrose/maltose (2) + H_2O (3) systems were correlated using the following nonlinear empirical eq 1[10]

$$w_1 = a + bw_2^{0.5} + cw_2 + dw_2^{2} + ew_2^{3} \qquad (1)$$

w_1 and w_2 represent the mass fraction of IL and sugar, respectively. The coefficients, a, b, c, d, and e, standard deviations (SD), and correlation coefficient (R^2) for the investigated systems are given in Table 2. On the basis of the obtained results, eq 1 can be satisfactorily used to correlate the binodal curve data of the investigated systems.

Tie-Lines. The tie-line data of [bmim]BF_4 (1) + sucrose/maltose (2) + H_2O (3) systems at different temperatures are shown in Table 3. The tie-lines of the two systems together with the binodal curves at T = 298.15 K, as an example, are clearly shown in Figures 3 and 4. The reliability of the tie-line

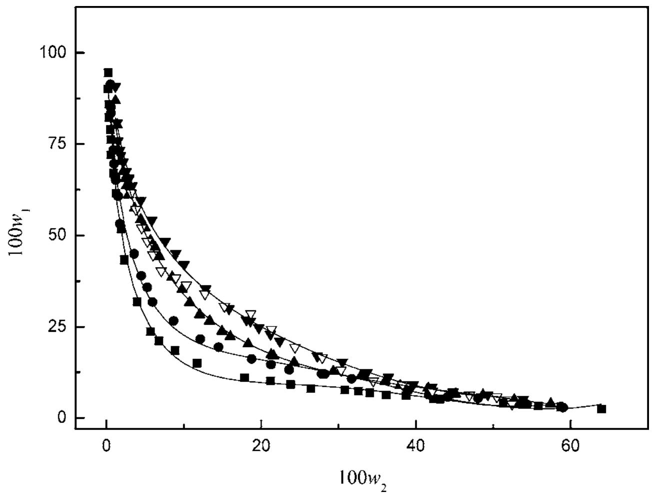

Figure 1. Binodal curves for $[bmim]BF_4$ (1) + sucrose (2) + H_2O (3) systems at different temperatures: ■, T = 278.15 K; ●, T = 288.15 K; ▲, T = 298.15 K; ▼, T = 308.15 K; ▽, literature data at T = 308.15 K;[11] the solid line represents the correlation according to eq 1.

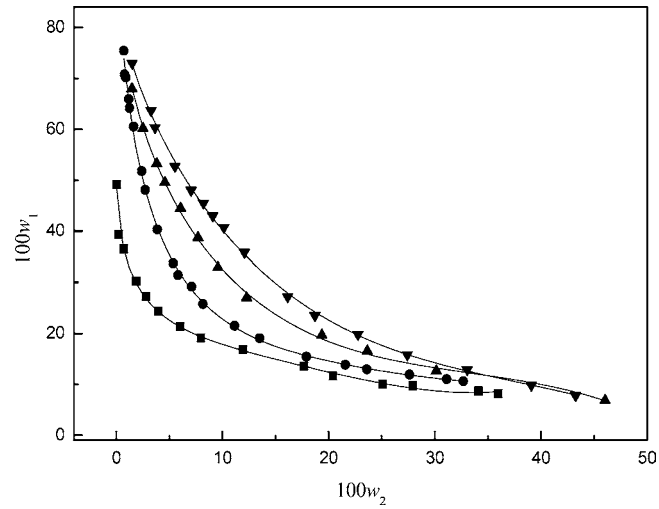

Figure 2. Binodal curves for $[bmim]BF_4$ (1) + maltose (2) + H_2O (3) systems at different temperatures: ■, T = 278.15 K; ●, T = 288.15 K; ▲, T = 298.15 K; ▼, T = 308.15 K; the solid line represents the correlation according to eq 1.

Table 2. Values of Parameters of Equation 1 for $[Bmim]BF_4$ + Sucrose/Maltose + H_2O at Different Temperatures

T/K	a	b	c	D	e	SD	R^2
			$[bmim]BF_4$ + Sucrose + H_2O				
278.15	116.8915	−62.5888	10.7118	−0.1179	0.0007	1.5435	0.9980
288.15	123.0778	−62.3395	10.9542	−0.1372	0.0009	2.4635	0.9937
298.15	116.4914	−38.0354	3.9149	−0.0152	0.0000	1.8400	0.9953
308.15	116.3306	−36.2986	4.3706	−0.0490	0.0004	2.2020	0.9941
			$[bmim]BF_4$ + Maltose + H_2O				
278.15	51.3856	−21.0656	4.0643	−0.0861	0.0010	1.0560	0.9950
288.15	113.0186	−53.2590	8.8254	−0.1094	0.0008	0.9077	0.9988
298.15	94.9674	−22.7744	0.51007	0.05176	−0.0007	0.45008	0.9993
308.15	90.8178	−12.9513	−1.5383	0.0636	−0.0006	0.6863	0.9992

Table 3. Tie-Line Data and Slopes of the Tie-Lines (STLs) for $[Bmim]BF_4$ (1) + Sucrose/Maltose (2) + H_2O (3) Systems at Different Temperatures

	IL-rich phase		sugar-rich phase			
T/K	100 w_1	100 w_2	100 w_1	100 w_2	STL	average of STL
			$[C_4mim][BF_4]$ (1) + Sucrose (2) + H_2O (3)			
278.15	63.02	0.85	12.80	23.20	−0.4201	
	73.50	0.75	10.50	26.40	−0.3946	
	81.91	0.63	8.80	30.80	−0.4071	
	88.90	0.32	7.21	35.18	−0.4267	−0.4122
288.15	62.97	1.20	13.23	25.22	−0.4829	
	73.32	0.87	12.14	29.63	−0.4701	
	78.84	0.73	9.86	32.33	−0.4581	
	84.02	0.61	7.53	37.98	−0.4885	−0.4749
298.15	52.63	4.83	17.43	22.40	−0.4991	
	62.72	2.48	14.02	26.92	−0.5018	
	73.12	1.43	11.62	32.94	−0.5124	
	80.23	1.38	8.93	37.99	−0.5135	−0.5067
308.15	65.83	2.27	17.18	28.27	−0.5344	
	73.52	1.63	13.33	34.70	−0.5494	
	80.80	1.38	9.47	42.07	−0.5474	
	88.83	1.16	6.15	50.88	−0.6013	−0.5581
			$[C_4mim][BF_4]$ (1) + Maltose (2) + H_2O (3)			
278.15	54.31	0.03	12.54	19.55	−0.4673	
	71.91	0.03	10.11	25.42	−0.4109	
	84.52	0.03	9.11	30.63	−0.4058	
	90.33	0.03	8.27	35.22	−0.4288	−0.4282
288.15	65.32	0.96	12.28	25.14	−0.4559	
	80.30	0.72	10.91	31.49	−0.4434	
	86.93	0.69	10.25	35.56	−0.4547	
	91.73	0.62	10.01	40.84	−0.4922	−0.4616
298.15	55.11	3.28	17.25	22.42	−0.5056	
	67.62	1.65	13.93	27.53	−0.4820	
	75.00	1.11	11.12	31.92	−0.4821	
	82.83	1.04	9.33	37.09	−0.4905	−0.4901
308.15	52.24	6.29	18.18	24.62	−0.5382	
	70.23	2.01	13.14	33.07	−0.5440	
	83.71	1.29	8.00	43.01	−0.5510	
	89.63	1.20	6.57	50.27	−0.5908	−0.5560

Table 4. Values of Parameters of Equations 2 and 3 for $[Bmim]BF_4$ (1) + Sucrose/Maltose (2) + H_2O (3) at Different Temperatures

T/K	k_1	n	k_2	r	R_1^2	R_2^2	SD_1	SD_2
				$[bmim]BF_4$ + Sucrose + H_2O				
278.15	0.0259	2.5984	0.0339	2.6451	0.9989	0.9987	0.0166	0.0218
288.15	0.0737	1.8856	0.0836	2.1398	0.9980	0.9997	0.0159	0.0057
298.15	0.1083	1.7051	0.1225	1.9245	0.9999	0.9994	0.0043	0.0123
308.15	0.1393	1.4595	0.1602	1.7606	0.9956	0.9950	0.0303	0.0333
				$[bmim]BF_4$ + Maltose + H_2O				
278.15	0.0225	2.5847	0.0300	2.6975	0.9986	0.9991	0.0255	0.0197
288.15	0.0354	2.4769	0.0510	2.5282	0.9997	0.9994	0.0102	0.0142
298.15	0.0698	1.9922	0.0889	2.1663	0.9979	0.9996	0.0207	0.0079
308.15	0.1171	1.8317	0.1397	2.0760	0.9999	0.9997	0.0040	0.0121

compositions was ascertained by the equations given by Othmer–Tobias (eq 2) and Bancroft (eq 3),[26–28] as shown below,

$$\frac{1 - w_1^{\mathrm{I}}}{w_1^{\mathrm{I}}} = k_1\left(\frac{1 - w_2^{\mathrm{s}}}{w_2^{\mathrm{s}}}\right)^n \tag{2}$$

$$\frac{w_3^{\mathrm{s}}}{w_2^{\mathrm{s}}} = k_2\left(\frac{w_3^{\mathrm{I}}}{w_1^{\mathrm{I}}}\right)^r \tag{3}$$

w_1^{I} is the mass fraction of IL in IL-rich phase, w_2^{S} is the mass fraction of sugar in the sugar-rich phase, and w_3^{S} and w_3^{I} are the mass fractions of water in sugar-rich phase and IL-rich phase, respectively. k_1, k_2, n, and r are the adjustable parameters obtained by regression analyses. The values of fit parameters, k_1, k_2, n, and r, along with SD and R^2 are given in Table 4 (subscripts 1 and 2 mean for eqs 2 and 3, respectively). From Table 4, it can be concluded that eqs 2 and 3 can be satisfactorily used to correlate the tie-line data of the investigated systems.

Slope of Tie-Line. The STL is calculated according to

$$\mathrm{STL} = \Delta w(\mathrm{sugar})/\Delta w(\mathrm{IL}) \tag{4}$$

Δw (sugar) and Δw (IL) are the differences in the concentration of the corresponding component sugar or IL between the two phases. STLs with different temperatures and different systems are also shown in Table 3. Table 3 shows that the STL increases with increasing temperature. As temperature increases, the mutual solubility of two phases also increases, and an increase in the amount of at least one of phase-forming components is needed for phase separation. As a result, tie-line slope increases with the increasing temperature. From Table 3, it can be seen that the STLs of the two systems are quite similar to each other, that is, the mutual solubility of $[bmim]BF_4$-rich phase and sucrose/maltose-rich phase is very close. This phenomena agree with the conclusion that the two-phase area of $[bmim]BF_4$–sucrose–H_2O is quite similar to that of the $[bmim]BF_4$–maltose–H_2O system in the Binodal Curves section.

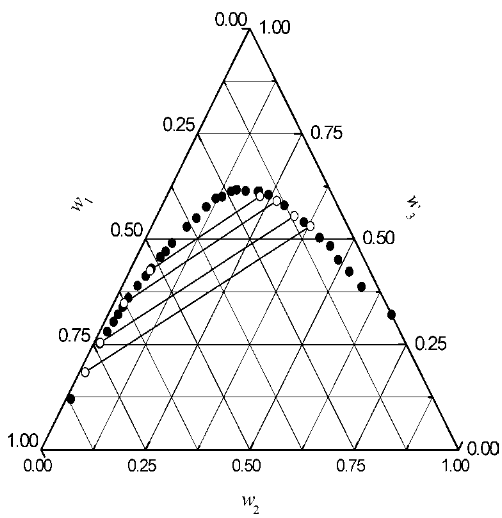

Figure 3. Ternary diagrams of $[bmim]BF_4$ (1) + sucrose (2) + H_2O (3) systems at T = 298.15 K: ●, binodal curve data; ○, tie-line data; the solid line represents tie-lines.

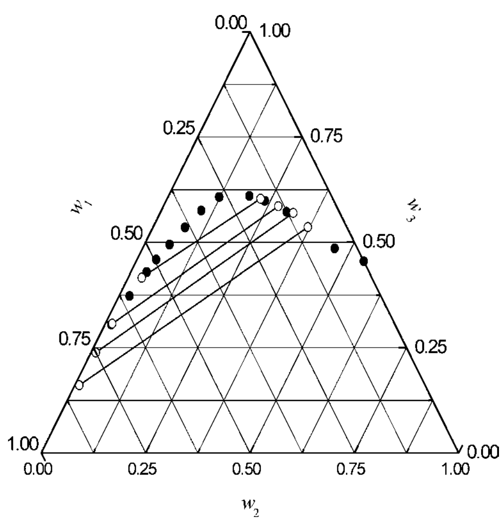

Figure 4. Ternary diagrams of $[bmim]BF_4$ (1) + maltose (2) + H_2O (3) systems at T = 298.15 K: ●, binodal curve data; ○, tie-line data; the solid line represents tie-lines.

Conclusions

The phase behaviors of IL-based ABSs, $[bmim]BF_4$ (1) + sucrose/maltose (2) + H_2O (3), were investigated in this research work. The binodal curves, tie-lines, and STLs were obtained at temperatures of T = (278.15, 288.15, 298.15, and 308.15) K. The binodal curve data were correlated using an empirical equation, and the tie-line data were correlated according to the Othmer–Tobias and Bancroft equations. Good agreements with the experimental data were obtained, and results showed that these equations can be satisfactorily used to correlate the experimental data of the investigated systems at the given temperatures.

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