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Isobaric Vapor–Liquid Equilibrium for Isopropanol + Water + 1-Ethyl-3-methylimidazolium Tetrafluoroborate

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Isobaric vapor–liquid equilibrium (VLE) data for the isopropanol (1) + water (2) system containing ionic liquid (IL) 1-ethyl-3-methylimidazolium tetrafluoroborate ($[EMIM]^+[BF4]^-$) at atmospheric pressure (101.32 kPa) were measured with a modified Othmer still. The results showed that the VLE of isopropanol + water in the presence of different contents of IL was obviously different from that of the IL-free system. The IL studied showed a salting-out effect, which gave rise to a change of the relative volatility of isopropanol and even to the elimination of the azeotropic point. It was found that the salting-out effect followed the order of $x_3' = 0.30 > x_3' = 0.20 > x_3' = 0.10$, which was probably attributed to the interaction between the ions resulting from the dissociation of IL and water.

Introduction

Special distillation processes, e.g., extractive distillation or salt distillation, are widely used in industry for azeotropic or close-boiling mixtures.[1,2] In extractive distillation, a third component, namely, the entrainer, is added to the mixture to alter the relative volatility and make the separation viable. In the salt distillation process, calcium chloride and potassium acetate are commonly used as entrainers, but they may erode the sieve plate, precipitate, and deposit in the tower due to their causticity and limited solubility. On the other hand, ionic liquids (often called room temperature ionic liquids, i.e., ILs) might be used as a suitable solvent salt and be superior to the commonly used entrainers due to their nonvolatility, less causticity, and good performance in improving the separation efficiency.[3] Furthermore, thermodynamic data for IL-containing systems are essential for a better understanding of the thermodynamic behavior of such systems, for separation design purpose, and for the development of thermodynamic models. Although some thermodynamic data for IL-containing systems have been reported, for example, VLE data,[4–7] LLE data,[8–13] GLE data,[14,15] and infinite activity coefficients of some organic solvents,[16–21] there are limited isobaric VLE data for such systems.

During the past years, isobaric or isothermal vapor–liquid equilibrium data for isopropanol + water containing salts, alkali, or even polymeric gel (vinyl alcohol + sodium acrylate copolymer)[22–27] have been reported. The equilibrium data for isopropanol and some other organic compounds[28–30] were also reported. However, to the best of our knowledge, there appears to be no VLE data on the ternary system of isopropanol + water containing 1-ethyl-3-methylimidazolium tetrafluoroborate. In this work, the isobaric VLE data for the ternary system of isopropanol + water + $[EMIM]^+[BF4]^-$ were measured at atmospheric pressure (101.32 kPa), and the effect of the IL on the separation performance of the isopropanol and water system is discussed.

Experimental Section

Chemicals. The chemical reagents used were ethanol, isopropanol, redistilled water, and IL. AR grade isopropanol and ethanol with a purity of above 99.7 % were purchased from Beijing Chemical Reagents Company, China. The purity of reagents was checked by gas chromatography (GC 4000A, China) and used without further purification. Distilled water was degassed and filtered using a 0.2 μm Millipore filter to remove dust. The IL, $[EMIM]^+[BF4]^-$, was provided by Chemical Engineering Research Institute of the Normal University of Hebei, with a mass fraction purity > 98 % observed by liquid chromatography. Furthermore, before the experiments, the IL was dried for 48 h at 363 K under a vacuum by the rotary evaporation to separate the IL from volatile byproducts and water. After experiments, the IL was reused after the rotary evaporation to eliminate the volatile components.

Apparatus and Procedure. Each solution was prepared gravimetrically using an electronic balance (Satorius, the uncertainty was about 0.1 mg).

The VLE data were measured by a circulation vapor–liquid equilibrium still (a modified Othmer still)[25,26] as shown in Figure 1. The total volume of the still was about 60 cm^3, of which about 50 cm^3 was occupied by the liquid solution. Energy was applied to the still through a heating rod controlled by an electric thermocouple. In the operation, a given liquid solution was put into the boiling chamber and heated. The vapor was condensed in the condenser and at the same time returned to the equilibrium chamber through the vapor-phase sampling port. Equilibrium was usually reached in about (0.5 to 1) h as indicated by the constant boiling temperature. The system was maintained in the equilibrium state for about 30 min, and samples of the vapor and liquid phase were taken from the sampling ports and then analyzed. The equilibrium temperature was measured by a precision and calibrated thermometer with an uncertainty of 0.1 K.

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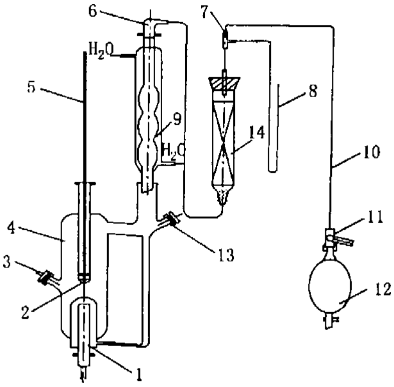

Figure 1. Experimental setup for VLE measurement: 1, heating bar; 2, glycerol; 3, liquid-phase sampling port; 4, equilibrium chamber; 5, thermometer; 6, ground joint; 7, three-way pipe; 8, U-style manometer; 9, condenser; 10, latex rubber tube; 11, three-way valve; 12, gas pressure ball; 13, vapor-phase sampling port; 14, desiccator.

Table 1. Vapor–Liquid Equilibrium Data for the Isopropanol (1) + Water (2) System at 101.32 kPa

T/K	x_1	y_1
357.15	0.072	0.487
356.13	0.110	0.511
354.75	0.250	0.519
354.75	0.299	0.540
353.80	0.422	0.550
353.80	0.440	0.560
353.35	0.560	0.616
353.20	0.572	0.621
353.30	0.818	0.784
353.94	0.905	0.866
355.07	0.992	0.988

The equilibrium compositions of the volatile components in the vapor and liquid phase were analyzed by gas chromatography. The gas chromatograph (GC 4000A) was equipped with a TCD detector. The chromatographic column (3 m × 0.3 mm) was packed with Porapak-Q. The carrier gas was hydrogen flowing at 30 $\text{cm}^3\cdot\text{min}^{-1}$, and the operating conditions were as follows: the injector and oven temperatures at 433 K and the detector temperature at 453 K. A calibration correction factor was obtained from a set of gravimetrically prepared standard solutions, which was used to quantify the amounts of isopropanol and water in the samples. In this way, the maximum uncertainty of the mole fraction of the components in the liquid and vapor phases was 0.003. The IL content in the liquid phase was determined using the gravimetric method by measuring the mass difference of liquid samples with and without IL.

Results and Discussion

To test the performance of the equilibrium apparatus, the binary vapor–liquid equilibria for the systems of ethanol + water and isopropanol + water were measured at 101.32 kPa. Our experimental data were in good agreement with those reported by Gmehling,[31] thus verifying that the experimental apparatus was reliable. The experimental results for the binary system of isopropanol (1) + water (2) are listed in Table 1 and compared to the literature in Figure 2.

Measurements were made for the ternary system of isopropanol (1) + water (2) + $[\text{EMIM}]^+[\text{BF4}]^-$ (3) by keeping the IL mole fraction constant in each set of the experiments. Results of isobaric VLE data for the isopropanol + water system containing IL $[\text{EMIM}]^+[\text{BF4}]^-$ at different IL mole fraction contents (from $x_3' = 10$ % to 30 %) are listed in Table 2. The x, y diagrams are also plotted in Figure 3. It should be mentioned that the mole fractions for the liquid components given in the tables and figures are on an IL-free basis.

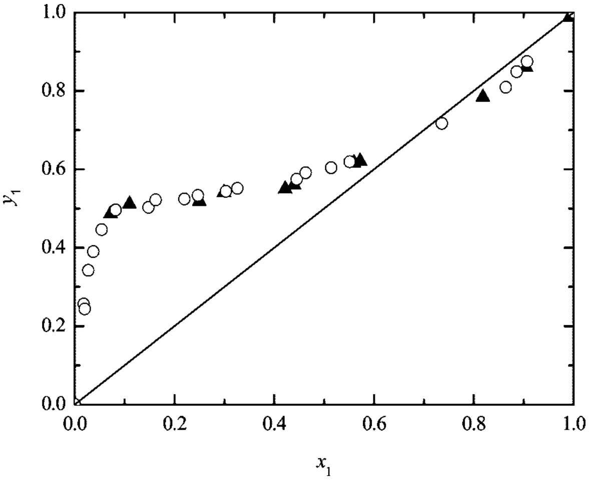

Figure 2. Isobaric VLE diagram for the binary system of isopropanol (1) + water (2) at 101.32 kPa: ○, ref 31; ▲, this work.

Table 2. Vapor–Liquid Equilibrium Data for the Ternary System Isopropanol (1) + Water (2) + $[\text{EMIM}]^+[\text{BF4}]^-$ (3) at 101.32 kPa

$100x_3'$	T/K	x_1	y_1	γ_1	γ_2	α_{12}
9.657	366.75	0.033	0.300	3.476	1.042	12.622
9.785	360.85	0.127	0.466	3.163	1.125	6.014
10.120	357.15	0.186	0.561	2.891	1.143	5.603
9.797	355.80	0.238	0.563	2.065	1.325	4.138
9.795	354.95	0.361	0.616	1.782	1.389	2.834
9.936	354.40	0.500	0.656	1.597	1.435	1.906
10.043	354.10	0.587	0.700	1.213	2.115	1.644
8.013	353.97	0.693	0.766	1.530	1.542	1.450
11.707	354.55	0.798	0.843	1.277	1.586	1.366
12.330	354.80	0.896	0.906	1.219	1.626	1.116
9.125	355.50	0.980	0.981	1.110	1.274	1.043
18.720	368.35	0.055	0.388	5.452	0.938	10.954
18.160	363.65	0.107	0.451	3.584	1.060	6.852
18.325	359.65	0.185	0.530	2.868	1.176	4.988
18.142	357.00	0.256	0.579	2.331	1.319	3.992
18.156	356.25	0.402	0.671	2.075	1.229	3.035
21.770	355.15	0.500	0.725	1.849	1.281	2.633
18.721	355.05	0.599	0.769	1.691	1.302	2.226
19.480	354.95	0.699	0.819	1.411	1.376	1.949
15.370	355.55	0.716	0.832	1.234	2.052	1.962
17.910	355.25	0.827	0.881	1.263	1.958	1.553
17.965	354.25	0.878	0.905	1.072	1.349	1.329
28.178	378.65	0.048	0.320	3.357	0.981	9.240
26.557	368.55	0.166	0.550	2.373	1.086	6.150
26.557	367.45	0.185	0.575	2.509	1.035	5.965
25.598	363.05	0.288	0.676	1.995	1.050	5.165
26.444	361.15	0.378	0.710	1.840	1.285	4.038
26.444	361.00	0.381	0.715	1.779	1.378	4.087
27.786	358.25	0.575	0.802	1.712	1.194	2.998
25.740	357.75	0.686	0.833	1.561	1.263	2.289
25.740	357.80	0.730	0.857	1.478	1.275	2.211
26.301	356.85	0.773	0.871	1.432	1.539	1.974
25.804	355.05	0.843	0.906	1.089	1.193	1.783
25.445	354.55	0.871	0.916	1.087	1.322	1.607
25.445	354.45	0.904	0.937	1.076	1.339	1.571

The effect of IL on the solution nonideality could be expressed by the activity coefficient of component i, γ_i, which could be calculated by the following equation:

$$\gamma_i = \frac{y_i \varphi_i P}{x_i' \varphi_i^{\mathrm{s}} P_i^{\mathrm{s}}} \tag{1}$$

where y_i represents the mole fraction of component i in the vapor phase; x_i' is the mole fraction of component i in the liquid phase (including IL); P the total pressure of the equilibrium system,

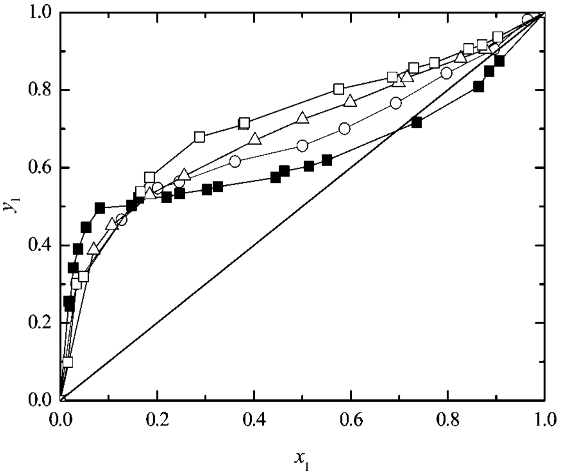

Figure 3. Isobaric VLE diagram for the isopropanol (1) + water (2) + $[EMIM]^+[BF4]^-$ (3) system at 101.32 kPa: ■, ref 31 ($x_3{}' = 0$); ○, $x_3{}' = 0.10$; △, $x_3{}' = 0.20$; □, $x_3{}' = 0.30$.

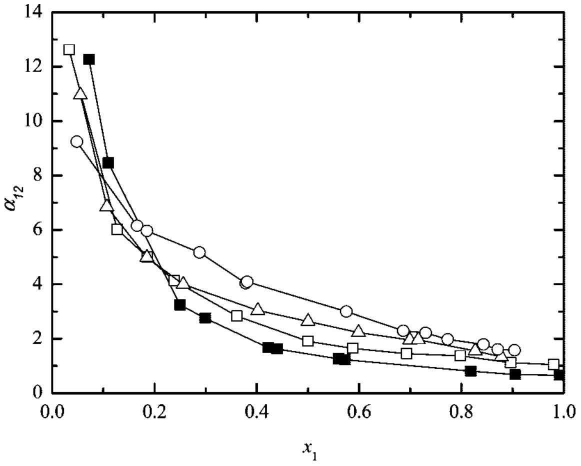

Figure 4. Relative volatility of isopropanol (1) + water (2) containing $[EMIM]^+[BF4]^-$ (3): ■, $x_3{}' = 0$; □, $x_3{}' = 0.10$; △, $x_3{}' = 0.20$; ○, $x_3{}' = 0.30$.

101.32 kPa; P_i^s is the vapor pressure of pure component i at the system temperature, which could be calculated by the Antoine equation using the Antoine constants from the literature;[28] φ_i is the fugacity coefficient of component i in the vapor mixture; and φ_i^s is the fugacity coefficient of pure component i in its saturated state. To simplify, the IL is treated as a nondissociating component, and the assumption of an ideal behavior is adopted for the vapor. The fugacity coefficients φ_i and φ_i^s are equal to unity at a low pressure.[28] Therefore, eq 1 could be rewritten as

$$\gamma_i = \frac{y_i P}{x_i{}' P_i^s} \tag{2}$$

It should be noted that IL does not appear in the vapor phase due to its nonvolatility. However, its mole fraction in the liquid phase is considered when calculating activity coefficients of isopropanol or water. In addition to the activity coefficient, the relative volatility of isopropanol to water is also calculated as follows

$$\alpha_{12} = \frac{y_1/x_1}{y_2/x_2} \tag{3}$$

where x_1 and x_2 are mole fractions of isopropanol and water, respectively, on an IL-free basis. The calculated activity coefficients and relative volatilities are also given in Table 2.

The IL investigated increased the relative volatility of isopropanol to water and thus showed a salting-out effect for isopropanol, as shown in Figures 3 to 5, where the fold lines are drawn to connect the points. Moreover, the azeotropic point at $x_1 = 0.6628$ for the isopropanol + water binary mixture is shifted upward with the addition of IL, and even the azeotropic phenomena could be totally eliminated at a specific IL content.

Figures 3 to 5 also indicate the complex effect of IL on the VLE of the isopropanol + water system. In the isopropanol-rich region, an increase of the IL content leads to a higher isopropanol content in the vapor phase and, therefore, to a larger relative volatility of the isopropanol. This is due to the strong selective interaction between IL and the water molecule. In contrast to the binary isopropanol + water mixture, the attractive interaction decreases the water activity and thus leads to an increased relative volatility of isopropanol. The IL is an organic molten salt, and its effect on the VLE of the isopropanol + water system may be ascribed to the affinity difference between ionic liquid and solute molecules. More specifically, the ions resulting from the dissociation of IL have a stronger attraction to water than to isopropanol due to the polar difference of ionic solvation energy, which leads to a preferential solvation of ions and enhancement of relative volatility of isopropanol. In the water-rich region, water molecules are preferentially "bonded" by ions. However, with an increase of IL content, for example, from $x_3{}' = 10$ % to 30 %, more and more isopropanol molecules are also bonded and thus the relative volatility of isopropanol to water decreases (at x_1 below 0.1).

To further investigate the salt effect of IL on isopropanol + water, relative volatilities of isopropanol to water were plotted in Figure 4 where the system contains 10 % to 30 % mole fraction of IL. With an increase in the IL component, the relative volatility of isopropanol to water increases (except for x_1 below 0.1). So the salting-out effect follows the order: 30 % > 20 % > 10 %.

The measured VLE containing the azeotropic system isopropanol + water and the IL $[EMIM]^+[BF4]^-$ are presented in a pseudobinary way in Figure 3, where the liquid-phase composition of the low-boiling component is the amount of this substance in the volatile part of the liquid phase. The IL component is demonstrated for each curve separately. The x, y diagram is shown in Figure 3, and the T, x, y diagram is shown in Figure 5. Figure 5 shows that the equilibrium temperatures increase at higher mole fractions of the IL, which indicates that it needs more heat energy to reach the new equilibrium at higher contents of the IL.

The addition of $[EMIM]^+[BF4]^-$ to this binary azeotropic mixture leads to a noticeable increase in the mole fraction of isopropanol in the vapor phase, breaking the azeotropic behavior of the system. This phenomena may be attributed to the interaction between water and $[EMIM]^+[BF4]^-$, which is stronger than the interaction between isopropanol and $[EMIM]^+[BF4]^-$, decreasing the water activity. This study confirms the capability of the IL as an entrainer for the separation of isopropanol + water.

Conclusions

ILs are becoming new alternative entrainers in special distillation processes. The isobaric VLE data for isopropanol + water containing $[EMIM]^+[BF4]^-$ were measured at atmospheric pressure (101.32 kPa). The results indicated that the IL studied showed a salting-out effect, which led to an elimination

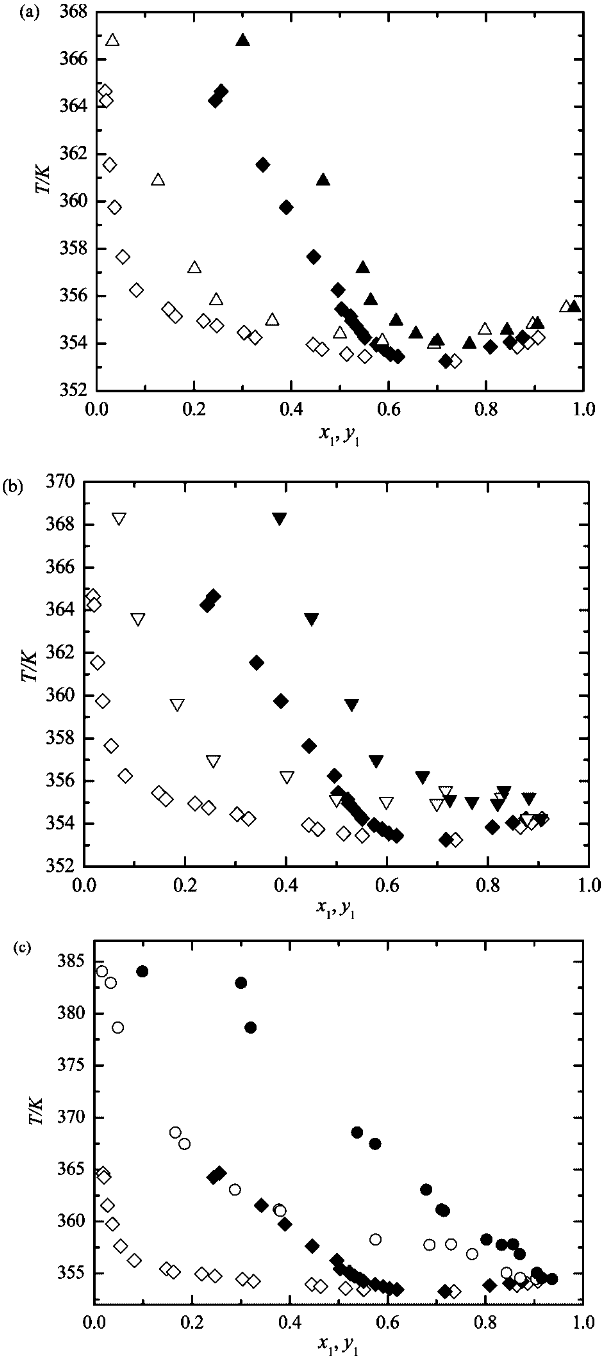

Figure 5. T, x, y diagram for the ternary system of isopropanol (1) + water (2) + [EMIM]$^+$[BF4]$^-$ (3) at different contents of IL: ◇, x_1 ($x_3' = 0$); ◆, y_1 ($x_3' = 0$); (a) △, x_1 ($x_3' = 0.10$); ▲, y_1 ($x_3' = 0.10$); (b) ▽, x_1 ($x_3' = 0.20$); ▼, y_1 ($x_3' = 0.20$); and (c) ○, x_1 ($x_3' = 0.30$); ●, y_1 ($x_3' = 0.30$).

of the azeotropic phenomenon at a specific IL content. Therefore, the results implied that [EMIM]$^+$[BF4]$^-$ is a promising additive for special distillation processes due to its notable salt effect and desirable properties, such as nonvolatility, nonflammability, and chemical stability. Moreover, because of its nonvolatility, the IL can be regenerated by different methods such as stripping, evaporating, or drying.

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