Isobaric Vapor−Liquid Equilibria for Water + Acetic Acid + Potassium Acetate

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Isobaric vapor−liquid equilibria for water + acetic acid + potassium acetate at different salt concentrations as well as for solvent + potassium acetate binary mixtures have been obtained at 100 kPa. Potassium acetate is more effective in the salting-out effect (on water) than lithium and sodium acetates. The eNRTL-CT model, which is a combination of electrolyte nonrandom two-liquid and chemical theory models, has been used to fit the experimental data. The binary parameters of the model have been obtained from the experimental binary vapor−liquid equilibria which have been used to suitably reproduce the vapor−liquid equilibria (VLE) for water + acetic acid + potassium acetate ternary system.

Introduction

The addition of salts to mixed-solvent mixtures is a common method for changing the relative volatility or modifying the azeotropic point in systems where separation by distillation is difficult. Even though most investigations have used saturated solutions in order to get the greatest salt effect, there is no doubt that for some systems the addition of small amounts of salt may improve the relative volatility of the solvents. Currently, the interactions between the species involved in these systems have been taken into account by using several models which are based on the local composition concept. Accordingly, most of these models constitute an effective tool to calculate liquid-phase activity in the presence of electrolytes. In a recent paper of Vercher et al.,[1] references of those models developed in the recent decades were presented.

However, these models are known to fail when acetic acid is present as one of the mixed solvents because its molecules associate by forming stable dimers. This is so because it constitutes an additional source of nonideality and makes it more difficult for chemical engineers to estimate VLE data. In fact, only three papers have used a predictive model in order to calculate and compare the VLE data[2−4] which confirms that the study of this kind of system represents a challenge for researchers.

The chemical theory model derived by Marek and Standart[5] and Marek[6] has been applied to account for the dimerization effect on both the liquid and gas phases. By use of the assumptions of the electrolyte nonrandom two-liquid (NRTL) and the chemical theory (CT) models, Vercher et al.[3] have obtained a combined model which demonstrated a suitable approach to predict the vapor−liquid equilibria of water + acetic acid + lithium acetate[3] and water + acetic acid + sodium acetate[4] ternary systems. These papers described both a salting-in effect and a small salting-out effect for water.

The aim of this work is to determine experimentally the effect of potassium acetate on isobaric vapor−liquid equilibrium of water + acetic acid at different salt concentrations and to check the electrolyte NRTL-CT model[3] with another mixed-solvent−salt system that presents association in the liquid and vapor phases. Likewise, the salt effect of potassium acetate will be compared with that produced by other alkaline acetates.

No isobaric data for this system are available in the literature. Only two papers concerning this ternary system have been found. Ciparis and Dzekciorius[7] studied the water + acetic acid + potassium acetate system at atmospheric pressure, but the pressure did not remain constant, varying from (99.06 to 103.32) kPa. In addition to this, Ciparis and Adomas[8] studied the same system at isothermal conditions of (313.15 and 333.15) K. The measured equilibrium pressure values at 313.15 K ranged from (4.93 to 7.20) kPa and from (13.20 to 19.20) kPa at 333.15 K, although the pressure accuracy was only 0.13 kPa.

Experimental Section

Materials. Glacial acetic acid 100% (Merck, PA grade) containing less than 0.2 mass % impurities, distilled water (Merck, HPLC grade), and potassium acetate (Aldrich, minimum purity 99.0 mass %) were used in this study. The potassium acetate was kept at 453 K in an oven at least for 24 h, whereas the solvents were directly used without further manipulation. The water content of acetic acid (maximum 0.03 mass %) and dissicated potassium acetate (maximum 0.07 mass %) was determined by the Karl Fischer method as it is explained in the sample analysis section.

Apparatus and Procedures. An all-glass dynamic recirculating still manufactured by Fischer was used to obtain experimental data. The equilibrium device is equipped with a Cottrell pump,[9] which makes it easier for both liquid and vapor phases to intimately mix. Pressures from (0.25 to 400) kPa and temperatures up to 523 K can be achieved with this still. A vacuum pump, a pressure sensor, and an electrovalve activated by an on−off pressure controller allowed us to keep pressure constant. The equilibrium pressure was measured with a digital manometer whereas a Pt-100 resistance thermometer was used to measure the boiling-point temperature. The standard uncertainties for pressure and temperature measurements were 0.01 kPa and 0.1 K, respectively.

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Table 1. Vapor−Liquid Equilibrium Data for Water (1) + Potassium Acetate (3) at 100 kPa

x_3	T/K	x_3	T/K	x_3	T/K	x_3	T/K
0.0064	373.15	0.0387	375.25	0.0698	377.95	0.1015	381.35
0.0109	373.45	0.0447	375.75	0.0752	378.45	0.1079	382.05
0.0157	373.75	0.0503	376.25	0.0801	378.95	0.1132	382.65
0.0210	374.05	0.0550	376.55	0.0854	379.55	0.1191	383.45
0.0265	374.35	0.0596	377.05	0.0904	380.15	0.1261	384.15
0.0326	374.85	0.0646	377.45	0.0957	380.75		

Every experimental point was obtained from an initial sample of water + potassium acetate or acetic acid + potassium acetate prepared with a Mettler AE 200 analytical balance (standard uncertainty of 10^{-7} kg) and afterward adding different quantities of acetic acid, water, or a mixture of water + potassium acetate. Equilibrium conditions were assumed when temperature and pressure stationary values were achieved for 30 min or longer.

Sample Analysis. Compositions of the condensed vapor phase (water + acetic acid) were analyzed by a Varian STAR 3400 CX gas chromatograph equipped with a thermal conductivity detector. The chromatographic column (2 m by $^1/_8$ in.) was packed with Porapak QS, and the flow rate of carrier gas, helium, was maintained at 40 $cm^3 \cdot min^{-1}$ (6.67×10^{-7} m^3 s^{-1}). The operating conditions were as follows: injector temperature, 493 K; oven temperature, 478 K; and detector temperature, 513 K. A calibration curve was obtained from a set of gravimetrically prepared standard solutions which was used to calculate the mole fraction of water in the unknown samples with a combined standard uncertainty of 0.001.

Liquid-phase samples of the ternary system, which were composed of water, acetic acid, and potassium acetate, were taken. The salt mass fraction was gravimetrically determined after separating the volatile components from a known mass of sample by evaporation to dryness, whereas its water content was measured with a Karl Fischer volumetric automatic titration (Metrohm, 701 KF Titrino). This titrator adds 10^{-9} m^3 steps of reagent until the analysis is finished, with the weight of the analyzed sample being changed according to its water content in order to improve the accuracy of the measurement. The one-component reagent used was Hydranal-Composite (Riedel-de Haën), while dried methanol (Riedel-de Haën, maximum 0.005 mass % water) was used as solvent. In addition to this, a buffer solution (Hydranal-Puffer from Riedel-de Haën) was used to stabilize the pH of the system provided that it is perturbed by the presence of acetic acid in the sample. In this way, the combined standard uncertainty of water, acetic acid, and potassium acetate mole fractions measurements in the liquid phase was 0.0015.

For binary systems (solvent + salt), the analysis of liquid phase was gravimetrically performed by means of evaporation to dryness. No analysis was made for the vapor phase. With this procedure, the salt mole fraction in the liquid phase was determined with a combined standard uncertainty of 0.0001.

Results and Discussion

Experimental Data. Vapor−liquid equilibria for the binary systems water (1) + potassium acetate (3) and acetic acid (2) + potassium acetate (3) as well as the vapor−liquid equilibrium for the water (1) + acetic acid (2) + potassium acetate (3) ternary system have been obtained at 100 kPa, and the results are reported in Tables 1−3. In these tables, x_3 represents the salt mole fraction in the liquid phase, x_1' is the mole fraction of water in the liquid phase, expressed on a salt-free basis, y_1 is the apparent mole fraction of water in the vapor phase, and T is the equilibrium temperature.

Table 2. Vapor−Liquid Equilibrium Data for Acetic Acid (2) + Potassium Acetate (3) at 100 kPa

x_3	T/K	x_3	T/K	x_3	T/K	x_3	T/K
0.0030	390.55	0.0337	392.15	0.0602	394.05	0.0943	397.75
0.0058	390.65	0.0370	392.35	0.0641	394.35	0.0989	398.35
0.0089	390.75	0.0401	392.65	0.0673	394.65	0.1034	398.85
0.0123	390.95	0.0426	392.75	0.0715	395.15	0.1082	399.55
0.0159	391.15	0.0460	393.05	0.0751	395.45	0.1127	400.15
0.0196	391.35	0.0485	393.15	0.0789	395.85	0.1184	400.75
0.0234	391.55	0.0511	393.35	0.0826	396.15		
0.0271	391.75	0.0542	393.65	0.0863	396.65		
0.0302	391.95	0.0571	393.85	0.0900	397.15		

Calculation of Phase Equilibrium. Marek's CT model[6] was applied to describe the deviation from ideality due to the dimerization of acetic acid in the vapor phase. The nonideality of the liquid phase is assumed to be a consequence of the interactions between solvent molecules and salt ions as well as the association of acetic acid in this phase. In this way, when the electrolyte NRTL model of Mock et al.[10,11] was used, the activity coefficients γ_1 and γ_2 include all sources[6] of liquid-phase nonideality. Hence, the physical meaning of these parameters is lost, with their values being different from those corresponding to a mixture of nonassociating solvents. Expressions for activity coefficients in ternary systems calculated by this procedure are reported elsewhere.[12] As far as the vapor phase is concerned, we have considered the dimerization of acetic acid molecules as the unique cause of nonideal behavior, which means determining the temperature dependence of the dimerization equilibrium constant. These are the basic assumptions of the eNRTL-CT model.

To represent the phase equilibrium of two solvents and an electrolyte system, nine binary adjustable parameters, which correspond to three solvent−solvent and solvent−salt pairs, must be determined. Of them, six are energy parameters ($\Delta g_{1,2}$, $\Delta g_{2,1}$, $\Delta g_{1,3}$, $\Delta g_{3,1}$, $\Delta g_{2,3}$, and $\Delta g_{3,2}$) and three are nonrandomness factors ($\alpha_{1,2} = \alpha_{2,1}$, $\alpha_{1,3} = \alpha_{3,1}$, and $\alpha_{2,3} = \alpha_{3,2}$).

The 1−3 and 2−3 binary solvent−salt parameters were determined from the experimental vapor−liquid equilibrium data given in Tables 1 and 2 by minimization of the objective function

$$F = \sum_j |T_{\text{exp}} - T_{\text{calc}}| \tag{1}$$

where T is the equilibrium temperature and the indices *exp* and *calc* denote the experimental and calculated values, with the summation being extended over the entirety of the data points. On the other hand, the 1−2 binary solvent−solvent parameters were taken from a previous paper.[3] The values of binary parameters and mean absolute deviations in vapor-phase composition and equilibrium temperatures are reported in Table 4.

To obtain the parameters given in Table 4, the equilibrium conditions of the eNRTL-CT model were considered, which allow us to calculate the equilibrium temperatures and vapor phase compositions by using the equations

$$Py_1Z_1 = X_1\gamma_1P_1^\circ \tag{2}$$

$$Py_2Z_2 = X_2\gamma_2P_2^\circ\Pi_2^\circ \tag{3}$$

where P is the total system pressure, y_i the apparent vapor-phase mole fraction of solvent i, X_i is the liquid-phase mole fraction of solvent i based on the assumption of total

Table 3. Vapor−Liquid Equilibrium Data for Water (1) + Acetic Acid (2) + Potassium Acetate (3) at 100 kPa

x_3	x'_1	y_1	T/K	x_3	x'_1	y_1	T/K	x_3	x'_1	y_1	T/K	x_3	x'_1	y_1	T/K
0.0105	0.006	0.006	390.75	0.0317	0.606	0.739	377.75	0.0728	0.007	0.005	394.55	0.0906	0.583	0.768	383.05
0.0103	0.102	0.178	386.35	0.0314	0.666	0.784	377.15	0.0724	0.088	0.143	391.45	0.0907	0.640	0.812	382.35
0.0105	0.183	0.290	383.45	0.0312	0.727	0.826	376.55	0.0725	0.162	0.256	389.35	0.0917	0.694	0.849	381.75
0.0106	0.258	0.378	382.15	0.0314	0.776	0.862	376.15	0.0726	0.239	0.365	387.25	0.0915	0.759	0.885	381.35
0.0105	0.329	0.458	380.65	0.0308	0.832	0.894	375.65	0.0721	0.319	0.468	385.45	0.0919	0.813	0.909	381.05
0.0104	0.410	0.540	379.35	0.0304	0.886	0.925	375.35	0.0720	0.386	0.549	384.25	0.0917	0.861	0.936	380.75
0.0104	0.479	0.605	378.35	0.0304	0.924	0.951	375.05	0.0717	0.453	0.621	383.15	0.0908	0.917	0.959	380.55
0.0104	0.545	0.668	377.45	0.0301	0.966	0.977	374.85	0.0717	0.516	0.688	382.15	0.0903	0.969	0.980	380.35
0.0103	0.613	0.724	376.65	0.0513	0.005	0.007	393.15	0.0714	0.585	0.747	381.15	0.1098	0.006	0.013	398.75
0.0103	0.672	0.771	376.05	0.0514	0.087	0.147	389.75	0.0713	0.647	0.797	380.45	0.1107	0.076	0.140	396.35
0.0103	0.720	0.813	375.45	0.0517	0.161	0.260	387.25	0.0705	0.709	0.837	379.95	0.1112	0.154	0.278	393.75
0.0102	0.775	0.850	374.95	0.0519	0.241	0.365	385.25	0.0703	0.772	0.873	379.35	0.1123	0.238	0.410	391.45
0.0102	0.830	0.883	374.45	0.0521	0.305	0.446	383.85	0.0699	0.826	0.902	378.95	0.1135	0.308	0.513	389.75
0.0100	0.888	0.918	374.05	0.0520	0.383	0.536	382.45	0.0703	0.869	0.928	378.65	0.1152	0.376	0.599	388.45
0.0100	0.925	0.947	373.85	0.0516	0.447	0.605	381.35	0.0691	0.924	0.955	378.35	0.1151	0.438	0.667	387.45
0.0099	0.969	0.976	373.55	0.0517	0.517	0.674	380.35	0.0679	0.972	0.980	378.25	0.1156	0.501	0.731	386.45
0.0320	0.006	0.007	391.75	0.0517	0.582	0.732	379.55	0.0861	0.009	0.008	396.65	0.1163	0.561	0.782	385.65
0.0320	0.096	0.165	387.85	0.0520	0.638	0.783	378.75	0.0869	0.090	0.151	393.25	0.1167	0.625	0.829	384.95
0.0320	0.181	0.283	385.25	0.0517	0.702	0.829	378.15	0.0877	0.162	0.269	390.85	0.1169	0.691	0.865	384.45
0.0318	0.264	0.386	383.25	0.0517	0.759	0.864	377.65	0.0901	0.244	0.393	388.85	0.1199	0.746	0.896	384.35
0.0318	0.345	0.479	381.65	0.0510	0.814	0.893	377.35	0.0896	0.319	0.494	387.15	0.1195	0.806	0.921	384.15
0.0316	0.414	0.553	380.45	0.0510	0.859	0.922	376.95	0.0902	0.380	0.566	386.05	0.1197	0.852	0.940	383.85
0.0316	0.489	0.629	379.45	0.0506	0.910	0.948	376.65	0.0902	0.449	0.642	384.95	0.1186	0.908	0.963	383.65
0.0318	0.542	0.686	378.55	0.0499	0.962	0.976	376.35	0.0905	0.522	0.715	383.75	0.1177	0.960	0.980	383.55

Table 4. Estimated Values of Energy Parameters, Δg_{ij} and Δg_{ji}, and Nonrandomness Factors, α_{ij}, for the eNRTL-CT Model and Mean Absolute Deviations, δy and δT, in Binary Systems

i component	j component	α_{ij}	Δg_{ij}/J·mol^{-1}	Δg_{ji}/J·mol^{-1}	δy^a	δT^b/K
water	acetic acid	1.476^c	3047^c	1464^c	0.003	0.03
water	potassium acetate	6.197	−908.5	2172		0.35
acetic acid	potassium acetate	0.175	−15346	35540		0.43

[a] $\delta y = (1/N)\ \Sigma\ |y_{\text{exp}} - y_{\text{calc}}|$. [b] $\delta T = (1/N)\ \Sigma\ |T_{\text{exp}} - T_{\text{calc}}|$. [c] Taken from Vercher et al.[3]

Table 5. Mean Absolute Deviations and Standard Deviations between Experimental and Calculated Values of the Vapor-Phase Mole Fractions and the Equilibrium Temperatures

	δy^a	σy^b	δT^c/K	σT^d/K
this work	0.014	0.018	0.42	0.50
Ciparis and Dzekciorius[7] (atm press)	0.012	0.018	0.51	0.70
Ciparis and Adomas[8] (T = 333.15 K)	0.016	0.018	0.75	0.88
Ciparis and Adomas[8] (T = 313.15 K)	0.047	0.022	0.82	1.27

[a] $\delta y = (1/N)\ \Sigma\ |y_{\text{exp}} - y_{\text{calc}}|$. [b] $\sigma y = [1/(N-1)]\ [\Sigma\ (y_{\text{exp}} - y_{\text{calc}})^2]^{1/2}$. [c] $\delta T = (1/N)\ \Sigma\ |T_{\text{exp}} - T_{\text{calc}}|$. [d] $\sigma T = [1/(N-1)]\ [\Sigma\ (T_{\text{exp}} - T_{\text{calc}})^2]^{1/2}$.

dissociation of electrolytes or, in case there is no electrolyte, it represents the apparent liquid-phase mole fraction of solvent i, γ_i is the activity coefficient of component i in the liquid phase, and $P_i°$ the vapor pressure of solvent i at equilibrium temperature. The vapor pressures of the pure solvent components were calculated by the Antoine equation using the parameters obtained from experimental vapor pressure values measured with the same recirculating still.[3] Furthermore, Z_i and $\Pi_2°$ can be viewed as correction factors for nonideality in the vapor phase and for the acetic acid vapor pressure, respectively, to take into account the acetic acid association, as it was already reported by Vercher et al.[3]

The nine binary parameters obtained were used to predict the vapor−liquid equilibrium of the water (1) + acetic acid (2) + potassium acetate (3) ternary system. In this way, the vapor-phase compositions and equilibrium temperatures for the same conditions as the experimental points given in Table 3 were calculated. The mean absolute and standard deviations presented in Table 5 indicate a good agreement between experimental and predicted points. In addition to this, Figure 1 shows the calculated and experimental vapor−liquid equilibria of the ternary system which is plotted on a $T-x_1'-y_1$ diagram for x_3 = 0, 0.052,

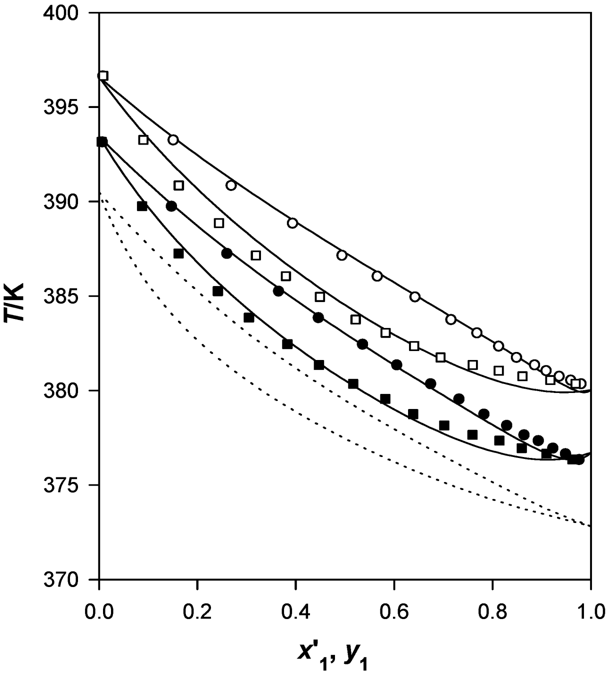

Figure 1. Experimental and calculated $T-x_1'-y_1$ diagram for water (1) + acetic acid (2) + potassium acetate (3) at 100 kPa: ■, $T-x_1'$ experimental, and ●, $T-y_1$ experimental (salt mole fraction, x_3 = 0.052); □, $T-x_1'$ experimental, and ○, $T-y_1$ experimental (salt mole fraction, x_3 = 0.091); solid line, $T-x_1'-y_1$ calculated; dotted line, $T-x_1'-y_1$ calculated salt-free system (calculation based on the eNRTL-CT model with the parameters from Table 4).

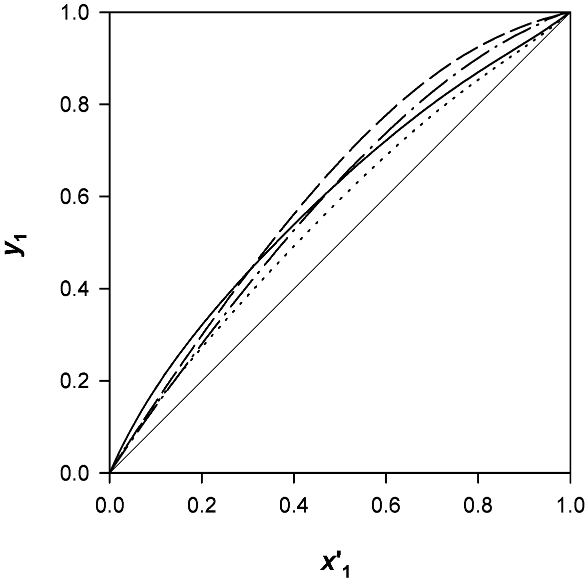

Figure 2. Calculated y_1-x_1' diagram for water (1) + acetic acid (2) + alkaline acetate (3) at 100 kPa and salt mole fraction x_3 = 0.091: dotted line, lithium acetate; dot-dashed line, sodium acetate; dashed line, potassium acetate; solid line, salt-free system.

and 0.091. In this figure, the predictive ability of the model can be observed. As expected, a similar behavior was achieved for the whole salt content studied.

Although data reported by Ciparis and Dzekciorius[7] and Ciparis and Adomas[8] could not be directly compared with those reported in this work because of the difference in experimental conditions, we were able to use the eNRTL-CT model to reproduce the vapor–liquid equilibria at conditions reported by these authors. In Table 5, the deviations between experimental and calculated values are presented. The results are similar to those obtained with sodium acetate.[4] The model adequately reproduces experimental data taken at atmospheric pressure although it seems to fail when isothermal data are considered.

To make comparisons, a y_1-x_1' diagram for water (1) + acetic acid (2) with different alkaline acetates (3) at x_3 = 0.091 is shown in Figure 2. Important differences in behavior can be observed in this figure. For instance, the addition of potassium acetate produces the maximum effect on relative volatility of water. Moreover, lithium acetate can only produce a salting-in effect on water, which is observed in the entire range of compositions,[3] whereas a salting-out effect is observed for sodium[4] and potassium acetates. However, according to Figure 2, this salting-out effect on water only appears at x_1' greater than 0.35 or 0.5 for potassium and sodium acetates, respectively, with this effect being more important for potassium acetate. Furthermore, potassium acetate gives the most significant rise in equilibrium temperature as well. Data from Figure 1 confirm that at x_3 = 0.091 and x_1' = 0.5 the increase in equilibrium temperature is 7.0 K, while for sodium and lithium acetates at the same conditions the increase in equilibrium temperature is (6.4 and 5.7) K, respectively.

Conclusions

The addition of potassium acetate to the water + acetic acid system produces a significant rise in equilibrium temperature, and an effect on the relative volatility of water greater than that produced by sodium and lithium acetates. The eNRTL-CT model has proved to be suitable to correlate the vapor–liquid equilibrium of the water + acetic acid + potassium acetate system. Experimental data agree well with the values obtained from the model throughout the range of compositions.

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