

Experimental study of phase equilibria in aqueous mixtures of phosphoric acid with isoamyl acetate and methyl isoamyl ketone at $T = (298.2, 308.2, \text{ and } 318.2) \text{ K}$

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ABSTRACT

This study demonstrates the experimental solubility and tie-line data for (water + phosphoric acid + isoamyl acetate or methyl isoamyl ketone) ternary systems at $T = (298.2, 308.2, \text{ and } 318.2) \text{ K}$ and atmospheric pressure for the first time. The concentration of each phase was determined by acidimetric titration, the Karl Fischer technique, and refractive index measurements. The experimental tie-line data were correlated using the UNIQUAC and NRTL models. The reliability of the experimental data was determined through the Othmer–Tobias and Hand plots. Distribution coefficients and separation factors were evaluated over the immiscibility regions.

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1. Introduction

Phosphoric acid (PA) is an important reagent or chemical intermediate in production of detergents, agricultural feeds, activated carbon, soaps, and several phosphate fertilizers [1,2]. In addition, to its traditional uses, the importance of this acid is quickly increasing in many other fields. It has been recently used as an additive for batteries and fuel cells [3,4]. It is well known that PA can be mainly produced by chemical wet and dry processes [5]. Thus, in the wet process method, the extraction of this acid from water is an important problem.

In the last decades, several important investigations have been carried out on the extraction and liquid–liquid equilibrium (LLE) measurements of the acid from aqueous solutions [6–17]. During the initial years of the 21st century, more equilibrium data for the aqueous mixtures of PA with organic solvents have been reported by several researchers [18–20]. To the best of our knowledge, ethers, ketones, alcohols, esters, and hydrocarbons have mainly been used as extractants in recovery of PA from water. Moreover, tributyl phosphate has been used as a solvent for extraction of PA from water [21–23]. However, further studies in this area are still important and needed for various industrial purposes.

We have recently reported the LLE data for the aqueous solutions of PA with various organic solvents at different temperatures

[24–27]. As a continuation of the previous works, we present the solubility and tie-line data for the ternary systems consisting of water, PA and organic solvents $T = (298.2, 308.2, \text{ and } 318.2) \text{ K}$. The selected organic solvents were isoamyl acetate (IAA) and methyl isoamyl ketone (MIK). In spite of structural similarity, however, they belong to two quite different chemical classifications. The UNIQUAC [28] and NRTL [29] models were used to correlate the experimental LLE data. For these equilibrium models, the values for the binary interaction parameters were obtained. In this work, distribution coefficients (D) and separation factors (S) were determined to establish the possibility of the use of these solvents for the separation process.

2. Experimental

2.1. Materials

Isoamyl acetate and methyl isoamyl ketone (stated mass fraction purity >0.99) were supplied from Merck. The molecular structures of these organic solvents are shown in Fig. 1. The chemicals were dried over molecular sieves (Merck 4Å). The stated purity of the chemicals was checked on the basis of their refractive indices and densities. Deionized water was further distilled before use. Analytical grade PA (H_3PO_4) containing 85 wt% was purchased from Merck. The acidimetric titration with 1N NaOH was used to confirm the purity of the acid. The measured refractive index and density data of the chemicals used in this study along with the literature values are listed in Table 1.

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Nomenclature

a_{ij}	optimized interaction parameter is expressed in K
b_{ij}	optimized interaction parameter is expressed in K
q	relative surface area per molecule
r	number of segments per molecule
n	number of tie-line
$rmsd$	root mean square deviation
w_{ijk}^{exp}	experimental mass fraction
w_{ijk}^{cal}	calculated mass fraction
T	absolute temperature (Kelvin)
u_{ij}	UNIQUAC interaction energy is expressed in $J\ mol^{-1}$
g_{ij}	NRTL interaction energy is expressed in $J\ mol^{-1}$

2.2. Apparatus and procedure

Details of the experimental apparatus, procedure and the tie-line measurements are similar to our previous work [26]. The solubility data of the ternary systems were determined by the cloud point method [30–32]. The visual measurements were repeated at least three times. The average of these readings was used for the component compositions (Table 2). Using the method of standard uncertainties [33], the uncertainty in the mass fraction of the solubility data was estimated to be better than ± 0.0008 .

The LLE measurements for the ternary systems were made at $T = (298.2, 308.2, \text{ and } 318.2)\text{ K}$. The temperature was estimated to be accurate to $\pm 0.1\text{ K}$ that was checked with a digital thermometer (Lutron TM-917). The mass fractions of the acid in both the phases were obtained by potentiometric NaOH titration. The water content of the organic layer was measured by the Karl Fischer method [34–36] using Metrohm-870 KF Titrino plus Karl Fischer titrator. The water content of the aqueous layer was determined using refractive index measurement method. The refractive indices of the prepared standard samples lying on the binodal curves were measured used for building standard curves (Table 3) [37]. The estimated uncertainty of all measured compositions is better than ± 0.001 . The uncertainty of the tie-line compositions was estimated according to the procedure suggested by Taylor and Kuyatt [33].

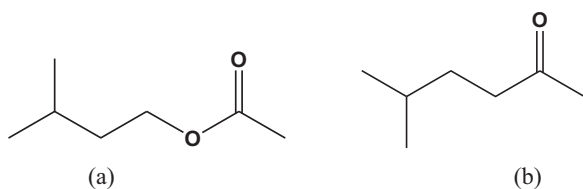


Fig. 1. The molecular structures of the solvents used in this study; (a) Isoamyl acetate (IAA), (b) Methyl isoamyl ketone (MIAK).

Table 1

Source, purity, refractive index (n) and density (ρ) of the pure components.^a

Chemical name	Supplier	Minimum mass fraction purity	n		ρ (kg m^{-3})	
			Exp.	Lit.	Exp.	Lit.
Isoamyl acetate (IAA)	Merck	>0.99	1.3975 ^b	1.398 ^b [44]	866.7 ^b	867.1 ^b [44]
Methyl isoamyl ketone (MIAK)	Merck	>0.99	1.4049 ^c	1.4062 ^c [41]	887.9 ^c	887.5 ^c [41]
Water (w)	Deionized and bidistilled		1.3324 ^b	1.3322 ^b [45]	997.0 ^b	997.08 ^b [45]

^a Uncertainties u are $u(n) = 0.0002$, and $u(\rho) = 0.001\text{ kg m}^{-3}$.

^b $T = 298.2\text{ K}$.

^c $T = 293.2\text{ K}$.

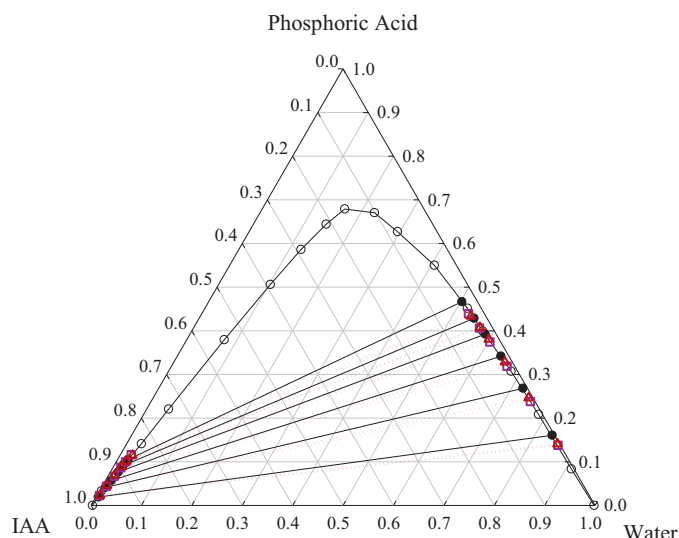


Fig. 2. Ternary phase diagram for LLE of [water (1) + PA (2) + IAA (3)] at $T = 298.2\text{ K}$; (○) experimental cloud points, (●) experimental tie-lines, (□) UNIQUAC calculated points, (△) NRTL calculated points ($\alpha = 0.3$).

3. Results and discussion

3.1. Experimental tie-line data

The LLE data for the (water + PA + IAA) and (water + PA + MIAK) ternary systems were experimentally determined at $T = (298.2, 308.2, \text{ and } 318.2)\text{ K}$ and atmospheric pressure. The experimental values for the ternary systems at each temperature are listed in Table 4. As can be seen the temperature effect on the binodal curves and biphasic region of the ternary systems is small at the temperatures studied. Typical LLE phase diagrams for the (water + PA + IAA) and (water + PA + MIAK) ternary systems at $T = 298.2\text{ K}$ are plotted and shown in Figs. 2 and 3, respectively.

To indicate the ability of the solvents (IAA and MIAK) in the extraction of the acid, distribution coefficients for water ($D_1 = w_{13}/w_{11}$) and PA ($D_2 = w_{23}/w_{21}$) and separation factor ($S = D_2/D_1$) were calculated from the experimental data. w_{13} , and w_{23} are the mass fractions of water and PA in the organic phase, respectively. w_{11} , and w_{21} are the mass fractions of water and PA in the aqueous phase, respectively. The experimental distribution coefficients and separation factors, for each system, are given in Table 5. The experimental data for the separation factor (S) are also compared with that of the correlated data. As can be seen from the table, the correlated values are relatively close to the experimental data for both models. The variation of experimental separation factor of PA as a function of the mass fraction of the acid in aqueous phase for (water + PA + IAA) and (water + PA + MIAK) systems are shown in Fig. 4a and b, respectively.

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