

Articles

Liquid–Liquid Extraction of Oxalic Acid from Aqueous Solutions with Tributyl Phosphate and a Mixed Solvent at 303.15 K[†]

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The phase diagram of the water + oxalic acid + tributyl phosphate ternary system was obtained at 303.15 K. Experiments were also conducted on the equilibrium distribution of oxalic acid between aqueous solutions and tributyl phosphate (TBP) + hexane (H) mixed solvent (with a 60/40 TBP/H percent volume ratio). Analysis of the results shows that the presence of a diluent (H) generally increases the selectivity when compared with pure TBP. This fact improves the extraction of oxalic acid from its aqueous solutions, but similar average distribution coefficients are obtained when the mixed solvent is used instead of pure TBP. A multistage cross-flow extraction process was performed in order to verify the accuracy of the basic equilibrium data and to obtain the number of stages required to extract oxalic acid from its aqueous solutions. This number was also graphically determined by using a distribution diagram in Bancroft's coordinates and analytically calculated by assuming virtual immiscibility between the feed and the extraction solvents.

Introduction

Removal of chemicals from aqueous effluents by conventional methods is generally an energy-expensive process, because these chemicals usually are present in low concentrations. Solvent extraction technology becomes increasingly important for the recovery or removal of organic substances (Juang and Huang, 1994; Kertes and King, 1986; Kiezyk and Mackay, 1973; King, 1992; Lo et al., 1983) for both energy savings and environmental protection.

Few studies of liquid–liquid equilibria (LLE) of aqueous solutions of oxalic acid can be found in the literature (Barnes et al., 1997; Faizal et al., 1990; Malmay et al., 1997; Scott et al., 1992; Zaheeruddin and Aslam, 1980). Since the solubility of oxalic acid in water is relatively low at 303.15 K (approximately 12% in mass fraction of anhydrous oxalic acid), the stock solution or the feed in the extraction process can never exceed this concentration. Consequently, it constitutes a relatively diluted solution so that a solvent extraction process could be used as a convenient way to remove oxalic acid from wastewaters.

Ternary aqueous oxalic acid systems using weak Lewis bases as solvents present low distribution coefficients and selectivities and were found to be inappropriate for extraction purposes (Barnes et al., 1997).

The aims of this paper are as follows: (i) to obtain the phase diagram of the water (W) + oxalic acid (AOA) + tributyl phosphate (TBP) ternary system at 303.15 K, analyzing the extractive properties of TBP (which is a

stronger Lewis base than those previously studied); (ii) to analyze the influence of the addition of a diluent (hexane, H) to TBP in order to reduce its viscosity and its density; (iii) to perform an extraction process (laboratory scale) to extract the oxalic acid from aqueous solutions with a mixed solvent (TBP/H), obtaining the number of stages necessary to achieve predetermined conditions for both feed and solvent using a multistage cross-flow process; (iv) to verify the applicability of an idealized extraction model which assumes that the feed solvent is virtually insoluble with the extraction solvent in order to calculate the required number of stages needed to obtain prefixed conditions and to compare these results with the experimental ones.

To favor mass transfer and phase separation, the viscosity should be as low as possible. Since TBP has a relatively high viscosity (3.069 mPa s at 303.15 K; see Table 2), it was mixed with H, and several quaternaries tie lines, W + AOA + TBP + H, at 303.15 K were obtained for each TBP/H ratio studied. Analyzing the experimental viscosity and density of both equilibrium phases and the distribution coefficients and selectivities, it was possible to find the best TBP/H ratio, trying to maintain the TBP contents as high as possible.

Among the different forms for performing an extraction process, a multistage cross-flow extraction was chosen. The number of theoretical stages (NTS) was evaluated both analytically (eq 1) and graphically, using a distribution diagram in Bancroft's coordinates (see Figure 3), and compared with experimental results. Once the best TBP/H ratio was obtained, the solvent ratio was determined from a single stage assay with aqueous oxalic acid solutions of three different compositions. Changing the solvent ratio and analyzing the oxalic acid extracted in each case, the solvent ratio range was obtained.

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Experimental Section

Materials. Bidistilled water, oxalic acid (Merck, p.a.), tributyl phosphate (Fluka, puriss. 99% GC), and hexane (Cicarelli, p.a.) were used. The purity of the oxalic acid was checked with standard sodium hydroxide (0.1 M) using phenolphthalein as indicator, with a result of better than 99%. Since their densities and viscosities compared well with literature values (within 0.1% for density, and 1% for viscosity), TBP and H were used without further purification.

Apparatus and Procedures. (a) Liquid–Liquid and Liquid–Solid Equilibria for the Ternary System. Isothermal solubility curves and tie lines were obtained simultaneously in the liquid–liquid region. Mixtures with compositions within the heterogeneous zone were vigorously shaken for at least 1 h at constant temperature of (303.15 ± 0.05) K. After decantation, the mass fractions of oxalic acid and water present in each liquid equilibrium phase were determined by titrating with standard sodium hydroxide (0.1 M) with phenolphthalein as indicator and by Karl Fischer titration (using a Mettler DL 18 Karl Fischer Titrator, Hydra Point Titrant 5 and Hydra Point Solvent G, Baker Analyzed Reagent), respectively. The errors due to acid–base and water titrations were generally accurate to within 1% and 0.5%, respectively.

Solid–liquid saturation curves and invariant points were obtained from saturated solutions in the L + S and 2L + S regions, respectively. Their liquid phases were analyzed with the same analytical procedure used for the liquid–liquid equilibrium.

(b) Determination of the Best TBP/H Ratio. Mixtures with volume ratios TBP/H of 20/80, 40/60, 60/40, and 80/20 were prepared, and their densities and viscosities, measured at (303.15 ± 0.05) K. Several quaternary tie lines in the L + L region were obtained for each TBP/H ratio studied, and the viscosity, density, and oxalic acid and water concentrations for both conjugated phases were determined. Densities were measured using a KEM, DA-300 density meter with an accuracy of $\pm 0.1 \text{ kg m}^{-3}$, while viscosities were measured with a Cannon-Fenske viscometer (calibrated with bidistilled water and benzene) with an accuracy of $\pm 0.01 \text{ mPa s}$. The experimental values of the difference of the densities between both phases, the viscosities of each phase, the distribution coefficients, and the selectivities were the parameters used to obtain the best TBP/H ratio.

(c) Solvent Ratio Determination. To determine the solvent ratio, $S_r = S/F$, we used a 50 mL separating funnel provided with a jacket for circulating water from a bath at (303.15 ± 0.05) K. Here S is the mass extraction solvent (TBP/H), while F is the mass feed solvent (W). After thermal equilibrium was reached, the separating funnel was manually stirred every 10 min during 30 min approximately. Three feed solutions (2, 7, and 12% in mass of anhydrous oxalic acid) were tested in a single contact extraction. The solvent ratios were varied between 0.2 and 1.0 with a step length of 0.2. Higher values than 1.0 were considered inappropriate for extraction purposes. In each extraction, the separating funnel was fed taking into account the desired solvent ratio value. After decantation, the oxalic acid present in each phase was determined with the same procedure used for liquid–liquid equilibrium in order to calculate the oxalic acid extracted for each pair feed concentration–solvent ratio studied.

(d) Multistage Cross-Flow Extraction Process. For the determination of the number of stages in this process (laboratory scale), we adopt as a final condition a difference

Table 1. Liquid–Liquid and Liquid–Solid Equilibrium Compositions, Distribution Coefficients (m), and Selectivities (β) for the Water (1) + Oxalic Acid (2) + Tributyl Phosphate (3) Ternary System at (303.15 ± 0.05) K

(a) Liquid–Liquid Equilibrium					
aqueous phase (R)		organic phase (E)		m	β
$100 w_{1R}$	$100 w_{2R}$	$100 w_{1E}$	$100 w_{2E}$		
99.5	0.0	5.3	0.0		
96.9	2.1	5.3	4.1	2.0	36.4
93.9	5.0	5.1	6.6	1.3	24.2
90.9	8.1	4.9	8.7	1.0	20.1
87.2 ^a	11.8 ^a	4.5 ^a	9.8 ^a	0.8	16.1

(b) Liquid–Solid Equilibrium			
aqueous phase		organic phase	
$100 w_{1R}$	$100 w_{2R}$	$100 w_{1E}$	$100 w_{2E}$
87.7	12.3	2.8	9.7
		0.9	10.8
		0.0	14.6

^a Invariant points in the 2L + S region.

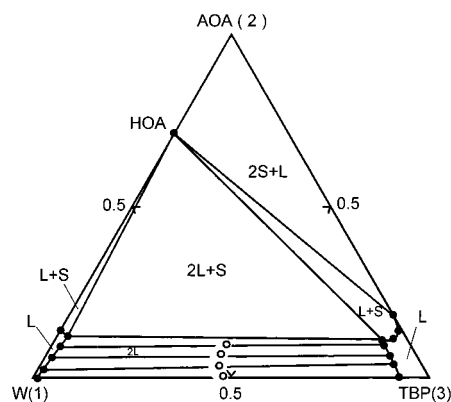


Figure 1. Equilibrium diagram for the ternary system water (W) + oxalic acid (AOA) + tributyl phosphate (TBP) at (303.15 ± 0.05) K: S = solid; L = liquid; ○ = global composition of the tie lines. HOA, hydrated oxalic acid.

of the percentage of oxalic acid between two successive stages lower than 1.5. The experimental results showed that, for the stock solution containing 2% of anhydrous oxalic acid, only one stage was necessary to reach that condition. A procedure similar to that employed for the solvent ratio determination was used, but in this process equal quantities of fresh solvent (TBP/H, with the appropriate ratio) were added to each raffinate from the previous stage, until the final condition was reached. On the other hand, the experimental determination of the number of stages necessary to exhaust the solvent for a given feed was performed with the same procedure, but equal volumes of feed were added to the extract phase of each stage. Oxalic acid concentrations were determined by the previous method for each extract, and the percentage of total oxalic acid extracted was obtained by mass balance.

Results and Discussion

Experimental equilibrium liquid–liquid and liquid–solid compositions for the ternary system water (W) + oxalic acid (AOA) + tributyl phosphate (TBP) at (303.15 ± 0.05) K are shown in Table 1, where the mass fraction compositions were estimated to an accuracy within ± 0.001 . Distribution coefficients and selectivities are also shown.

Figure 1 shows the equilibrium diagram for this ternary system at 303.15 K. Since this phase diagram includes solid

Table 2. Densities (ρ) and Viscosities (η) of Tributyl Phosphate (TBP) + Hexane (H) Binary Mixtures at (303.15 \pm 0.05) K

TBP/H ratio (v/v)	ρ (kg m ⁻³)	η (mPa s)	TBP/H ratio (v/v)	ρ (kg m ⁻³)	η (mPa s)
100/0	968.0	3.07	40/60	777.8	0.77
80/20	906.8	1.68	20/80	713.7	0.38
60/40	841.3	1.15	0/100	650.4	0.26

phases, it might belong to type 4 in the classification proposed by Treybal (1963).

Oxalic acid distribution coefficient, m , is defined as the ratio of the acid mass fraction in the organic phase (w_{23}) to that in the aqueous phase (w_{21}). The selectivity (β) is another important property, defined as $\beta = mw_{11}/w_{13}$, where w_{11} and w_{13} are the mass fractions of water in the raffinate phase and in the extract one, respectively. Table 1 shows the distribution coefficients and selectivities for the water + oxalic acid + tributyl phosphate ternary system at 303.15 K. The values are sufficiently high to consider TBP as a potentially useful solvent for the extraction of oxalic acid from wastewaters. However, its viscosity is relatively high for extraction purposes because it was mixed with hexane. Solutions with 20/80, 40/60, 60/40, and 80/20 TBP/H volume ratios were prepared and their densities and viscosities determined as previously described. Table 2 shows these last two experimental values at (303.15 \pm 0.05) K.

It is well-known that solvents with high viscosities produce stable aqueous emulsions which are inadequate for extraction processes. The viscosity of the mixed solvent and the two phases need to be close to that of water so that no special equipment is required. Furthermore, the difference of densities between the two equilibrium phases should be greater than 50 kg m⁻³ (*Ullmann's Encyclopedia of Industrial Chemistry*, 1988).

Since only water and oxalic acid were analytically determined in each conjugated phase for the water + oxalic acid + tributyl phosphate + hexane quaternary system, the mixed solvent concentration was obtained taking into account that the sum of the percentages of all components must be equal to 100. Surely, solvent (TBP) and diluent (H) are distributed dissimilarly between conjugate phases and their ratio in each phase should be different from the initial value. This fact was not a problem because we were not interested in obtaining the quaternary phase diagram but only in analyzing the influence of H on the extraction properties for each of the quaternary sectional planes. A similar quaternary system was studied by Malmay et al. (1997) using dodecane instead of hexane as diluent. However, an error was committed in their paper because they plotted the ends of the quaternary ties lines resting on the solubility curve, similarly to what happen in ternary systems.

Table 3 shows the experimental equilibrium data for the different TBP/H ratios studied. Densities and viscosities of each conjugate phase are also shown, together with the distribution coefficients and selectivities.

Table 3 also shows that the quaternary systems with TBP/H ratios of 40/60 and 60/40 are solutropics. Only two TBP/H ratios (80/20 and 60/40) have distribution coefficients and selectivity values compatible with an extraction process, but the viscosity of the extract phase for the 80/20 ratio generally has a higher value than pure TBP. Therefore, the best ratio among those studied to extract oxalic acid from aqueous solutions is 60/40 of TBP/H. The difference of densities between the conjugated phases is

always higher than 50 kg m⁻³ (*Ullmann's Encyclopedia of Industrial Chemistry*, 1988).

To extract this acid by using a liquid-liquid extraction process, a fundamental variable is the solvent ratio. Its best value was obtained by analyzing the percentage of oxalic acid extracted as a function of the solvent ratio in a single-contact operation. Figure 2 shows that the percentage of extracted acid increases when the solvent ratio is increased.

An extraction lower than 25% in the first stage was considered inadequate, because lower values would lead to a high number of stages. Figure 2 shows that solvent ratios of 0.2 and 0.4 do not achieve this condition for 7 and 12% of oxalic acid concentration in the feed, while for 2% the best solvent ratio is 0.4. On the other hand, a solvent ratio equal to 1 produces little increment in the percentage of oxalic acid extracted for the two higher concentrations of the feed. We choose therefore the range 0.6-0.8, to perform the experimental determination of the number of stages with feeds having 7 and 12% of anhydrous oxalic acid. The total percentage of oxalic acid removed was between 80 and 90% for all the combinations of feed and solvent ratios studied.

Table 3 shows that the selected TBP/H ratio and water are virtually immiscible. Therefore, a simplified procedure can be used to calculate the number of theoretical stages (NTS) for this extraction process. If the distribution coefficient $K' (=Y_i/X_i)$, where Y_i and X_i are the mass ratios of AOA to TBP/H in the extract product and in the raffinate one for the i th tie line in Bancroft's coordinates, respectively, the ratio of the masses of the extraction solvent (S) and the feed solvent (F) are constant and fresh solvent is used as the extraction solvent, the NTS in a multistage cross-flow extraction required to achieve a predetermined raffinate concentration can be calculated as follows (Perry et al., 1992):

$$\text{NTS} = \log(X_F/X_N)/\log(K'S/F + 1) \quad (1)$$

where X_F and X_N are the mass ratio of AOA to W in the feed and the mass ratio of AOA to TBP/H in the final raffinate product in Bancroft's coordinates, respectively. Taking into account that the raffinate and extract produced in each stage are in equilibrium, an oxalic acid mass balance for the i th stage gives

$$Y_i = (F/S)(X_i - X_{i-1}) \quad (2)$$

Figure 3 shows the distribution curve and the operating lines described by eq 2. This curve was drawn by plotting all experimental tie lines (Table 3 for 60/40 of TBP/H) and the equilibrium values obtained in the solvent ratio determination, with all operating lines having equal slopes ($=F/S$).

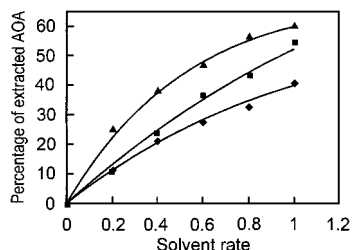
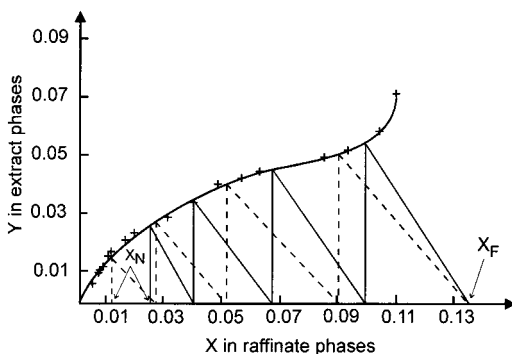
The last stage gives the compositions of the final extract and raffinate phases, which must satisfy the final experimental conditions.

Table 4 shows the number of stages obtained experimentally, analytically using eq 1, and graphically (Figure 3), for the quaternary system W + AOA + TBP + H at 303.15 K. It also shows that the number of theoretical stages obtained graphically is similar to the experimental ones, which is an indication that the equilibrium was achieved in each stage and the assumption that the feed solvent (W) and the extraction solvent (TBP/H) are virtually insoluble over the range of solute concentrations involved was correct.

Figure 3 shows that K' is not constant, since the equilibrium curve is not exactly a straight line. However,

Table 3. Experimental Equilibrium Data for the System: Water (1) + Oxalic Acid (2) + Tributyl Phosphate (3) + Hexane (4) for the Different TBP/H Volume Ratios, Densities (ρ), and Viscosities (η) of Each Conjugated Phase, Distribution Coefficients (m), and Selectivities (β) at (303.15 \pm 0.05) K

aqueous phase (R)				organic phase (E)				m	β
100 w_{1R}	100 w_{2R}	$\rho/(\text{kg m}^{-3})$	$\eta/(\text{mPa s})$	100 w_{1E}	100 w_{2E}	$\rho/(\text{kg m}^{-3})$	$\eta/(\text{mPa s})$		
TBP/H = 80/20									
99.40	0.00	997.5	0.81	3.80	0.00	906.9	1.22		
98.80	0.94	999.9	0.81	3.70	2.00	921.6	2.41	2.1	56.8
96.50	2.55	1007.5	0.83	3.79	4.14	932.1	3.26	1.6	41.3
94.25	4.01	1015.8	0.85	4.04	6.20	942.6	3.87	1.5	36.1
92.36	4.85	1023.4	0.88	4.42	7.58	944.1	3.96	1.5	32.6
TBP/H = 60/40									
97.80	0.00	994.3	0.79	2.30	0.00	841.3	1.13		
97.50	0.92	999.1	0.80	2.90	1.31	841.1	1.08	1.4	47.9
97.31	1.08	999.9	0.81	2.98	1.52	844.1	1.12	1.4	45.9
96.47	1.79	1003.6	0.82	2.93	2.19	854.3	1.25	1.2	40.3
93.37	4.73	1019.0	0.87	2.76	4.20	879.4	1.54	0.9	30.4
TBP/H = 40/60									
98.55	0.00	1000.5	0.82	1.45	0.00	788.8	0.65		
93.58	1.35	1003.4	0.82	1.62	1.87	795.0	0.75	1.4	80.0
90.89	4.72	1017.1	0.87	1.47	2.86	798.6	0.79	0.6	37.5
88.87	6.85	1028.4	0.91	1.52	3.36	800.1	0.85	0.5	28.7
87.32	8.10	1038.5	0.96	2.24	4.14	802.7	0.88	0.5	19.9
TBP/H = 20/80									
98.00	0.00	999.2	0.81	1.00	0.00	718.1	0.40		
95.43	2.31	1006.1	0.83	0.91	0.53	721.4	0.41	0.2	24.1
92.00	5.71	1021.9	0.89	0.88	1.57	727.4	0.44	0.3	28.7
89.76	8.37	1034.1	0.94	1.20	2.12	725.0	0.45	0.2	18.9
87.87	9.76	1045.5	0.99	1.54	2.87	726.4	0.46	0.3	16.7

**Figure 2.** Percentage of extracted oxalic acid as a function of the solvent ratio. Feed: \blacklozenge 12%; \blacksquare 7%; \blacktriangle 2% of anhydrous oxalic acid.**Figure 3.** Multistage cross-flow extraction with immiscible solvents: \times , experimental equilibrium distribution curve in Bancroft's coordinates; — and - -, operating lines for solvent ratios $S_1 = 0.6$ and 0.8, respectively.

the number of theoretical stages obtained analytically with eq 1 is in fairly good agreement with the experimental results because the influence of this hypothesis on eq 1 surely has lesser importance than that related with the nonmiscibility of phases.

For the experimental determination of the number of stages necessary to exhaust the solvent for a given feed and solvent ratio, three stages were always necessary to obtain a difference of percentage of oxalic acid between two successive stages lower than 1.5.

Table 4. Number of Stages Obtained Experimentally, Theoretically Using Equation 1, and Graphically Using Bancroft's Coordinates to Obtain a Final Raffinate Concentration (X_N)

solvent ratio	X_N	exptl	theoretical	graphically
Feed: 12% of Anhydrous Oxalic Acid				
0.6	0.0229	4	3.8	4.1
0.8	0.0124	4	4.1	4.0
Feed: 7% of Anhydrous Oxalic Acid				
0.6	0.0190	3	3.2	3.0
0.8	0.0113	3	3.5	3.1

Conclusions

The equilibrium diagram for the ternary system water + oxalic acid + tributyl phosphate was obtained at (303.15 \pm 0.05) K.

Analyzing the distribution coefficients and selectivities, we conclude that TBP appears to be a good solvent for extraction purposes when oxalic acid needs to be extracted from its aqueous solutions, because the distribution coefficients are always higher than 1 and the selectivities are sufficiently high (see Table 1). However, the viscosity of TBP needs to be reduced in order to avoid an excessive energy consumption and to improve phase settling. This was made mixing it with hexane which decreases the viscosity value from 3.069 mPa s for pure TBP to 1.151 mPa s for the selected TBP/H ratio (see Table 2).

Similar average distribution coefficients were obtained for the ternary system (W + AOA + TBP) and the quaternary system (W + AOA + TBP + H) with a TBP/H ratio of 60/40 (1.275 and 1.225, respectively; see Tables 1 and 3). But the average selectivities are higher for the quaternary system than for the ternary one (41.1 and 24.2, respectively; see Tables 1 and 3), improving the extraction of oxalic acid from its aqueous solutions as compared to pure TBP.

The number of theoretical stages obtained graphically was similar to the experimental value, which confirms that the equilibrium in each stage was achieved, and that the

hypothesis of virtual immiscibility between the feed (W) and the extraction (TBP/H) solvents is correct. The little deviation of the number of theoretical stages obtained analytically (eq 1) is due to the variation of the distribution coefficient (K) for this system.

Finally, an oxalic acid free extract phase could be obtained by adding calcium hydroxide and filtrating the precipitate calcium oxalate.

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