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Liquid−Liquid Equilibrium in the System Phosphoric Acid/Water/Tri-*n*-butyl Phosphate/Calcium Chloride

Yang Jin,[†] Jun Li,* Jianhong Luo,[†] Dongsheng Zheng,[†] and Li Liu[†]

Department of Chemical Engineering, Sichuan University, Chengdu, Sichuan, 610065, People's Republic of China

Liquid−liquid equilibrium and tie line data in the system phosphoric acid/water/tri-*n*-butyl phosphate/calcium chloride has been determined at 298.15 K, in the concentration range from $w = 0.028$ to 0.20 phosphoric acid and $w = 0$ to 0.25 calcium chloride. Distribution coefficients of phosphoric acid and separation factors of water and calcium chloride were determined to establish the extracting capability of tri-*n*-butyl phosphate. The results show that tri-*n*-butyl phosphate can serve as an adequate extractant to recover phosphoric acid from dilute phosphoric acid containing calcium chloride.

Introduction

Phosphoric acid is a widely used basic raw material in many areas such as fertilizers, food additives, and detergents. Conventionally, phosphoric acid is manufactured by decomposing phosphate rock with sulfuric acid. But this process suffers from the fact that a large amount of gypsum is produced and utilization of gypsum is difficult and expensive.[1] These problems can be solved by dissolving phosphate rock with hydrochloric acid.[2] In the hydrochloric acid route, the middle or low grade phosphate rock can be used to produce phosphoric acid with no gypsum byproduct. Besides, byproduct hydrochloric acid from the chloralkali industry can also be utilized properly in the hydrochloric acid route.

Many efforts have been made to develop a commercially feasible process using hydrochloric acid instead of sulfuric acid for recovery of phosphorus from phosphate rock. The IMI (Israel Mining Industries) process was invented in 1957[3] and has been implemented since the early 1960s in a few plants. In this process, phosphate rock is decomposed by hydrochloric acid. After separation of the insoluble residues, a solvent extraction technique is applied to extract phosphoric acid from the leach solution which contains phosphoric acid, calcium chloride, and other impurities. The solvents used for extraction are low aliphatic alcohols, especially butyl alcohol and isoamyl alcohol.[4,5] Those solvents are usually volatile and slightly soluble in water, so recovery of the extractant is necessary, but raises the cost of phosphoric acid. Besides, the low flash points of low aliphatic alcohols may cause safety problems in manipulation. Therefore, it is of significance to search for a new and suitable solvent for the extraction of phosphoric acid from the leach solution.

Tri-*n*-butyl phosphate (TBP) is widely used for the extraction of phosphoric acid[2,6−9] because of its immiscibility with aqueous solution, good selectivity to phosphoric acid, and easy recovery. Yet, phase equilibrium data in the system phosphoric acid/water/TBP/calcium chloride have not been reported. Also, the capability of TBP to extract phosphoric acid from dilute phosphoric acid containing calcium chloride has not been revealed.

The aim of this work is to study the phase equilibrium in the system phosphoric acid/water/TBP/calcium chloride at 298.15 K, in the concentration range from w (mass fraction) $= 0.028$ to 0.20 phosphoric acid and $w = 0$ to 0.25 calcium chloride. Distribution coefficients of phosphoric acid and separation factors of water and calcium chloride are determined from the tie-line data. The extracting capability of TBP to recover phosphoric acid from dilute phosphoric acid containing calcium chloride is estimated.

Experimental Section

Materials. TBP was provided by the Donghu Chemical Company, China and was used without any further purification. Phosphoric acid and calcium chloride were supplied by the Kelong Co., China. All of the reagents were analytical grade. Deionized water was used in the experiments.

Procedure. The experiments were carried out at $T = (298.15 \pm 0.1)$ K with a water thermostat. Known amounts of dilute phosphoric acid and TBP were mixed in a separatory funnel. The mixture was shaken for 2 h and then left to settle for 2 h to completely separate the two liquid phases. The aqueous sample was taken from the bottom opening of the separatory funnel, being taken carefully to leave a layer of aqueous solution at least 1 cm thick below the interface. Then the remaining aqueous layer as well as at least 1 cm thick of the organic phase in the separatory funnel was collected carefully in a 10 mL buret. The volume of the aqueous and organic phase in the buret was determined and recorded as $V^{\mathrm{a}}_{\mathrm{I}}$ and $V^{\mathrm{o}}_{\mathrm{I}}$. The organic solution left in the separatory funnel was taken as the organic sample. After separation, the aqueous and organic samples were weighed and recorded as $m^{\mathrm{a}}_{\mathrm{II}}$ and $m^{\mathrm{o}}_{\mathrm{II}}$. The densities of each phase ρ^{a} and ρ^{o} were measured using a method described elsewhere[10] with Ostwald−Sprenge-type pycnometers having a bulb volume of 10 cm^3 and an internal capillary diameter of about 1 mm. The total mass of each phase m^{a} and m^{o} can be calculated as $m^{\mathrm{a}} = m^{\mathrm{a}}_{\mathrm{II}} + \rho^{\mathrm{a}}V^{\mathrm{a}}_{\mathrm{I}}$ and $m^{\mathrm{o}} = m^{\mathrm{o}}_{\mathrm{II}} + \rho^{\mathrm{o}}V^{\mathrm{o}}_{\mathrm{I}}$. Then, the samples of each phase were analyzed. Phosphoric acid concentrations in both phases were determined by the quinoline phosphomolybdate gravimetric method.[11] Water concentrations in the organic phase were measured by the Karl Fischer method.[12] Calcium concentrations in both phases were determined by ethylenediaminetetraacetic acid (EDTA) titration using Solochrome black as

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* To whom correspondence should be addressed. E-mail: lijun@scu.edu.cn. Tel./fax: 0086-028-85460936.

[†] E-mail: Yang Jin: jinyangyoung@126.com; Jianhong Luo: ljh00308033@163.com; Dongsheng Zheng: dsh.zheng78@gmail.com; Li Liu: lilysure@163.com.

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Table 1. Liquid−Liquid Equilibrium Data in the System Phosphoric Acid (1)/Water (2)/TBP (3)/Calcium Chloride (4) at 298.15 K[a]

initial mixtures				aqueous phase					organic phase							
w_1	w_2	w_3	w_4	w_1	w_2	w_3	w_4	ρ^a(g·mL^{-1})	w_1	w_2	w_3	w_4	ρ^o(g·mL^{-1})	D_1	S_2	S_4
$CaCl_2$ = 0.0 in Dilute Phosphoric Acid																
1.46	49.86	48.68		2.83	97.05	0.12		1.0154	0.20	6.87	92.93		0.9729	0.07	1.0	
2.94	48.73	48.34		5.59	94.30	0.11		1.0294	0.50	6.81	92.70		0.9734	0.09	1.2	
4.31	47.44	48.26		8.04	91.85	0.11		1.0443	0.89	6.74	92.38		0.9750	0.11	1.5	
5.49	46.92	47.59		9.99	89.91	0.09		1.0602	1.28	6.69	92.02		0.9771	0.13	1.7	
7.38	45.43	47.19		12.83	87.07	0.10		1.0752	2.30	6.64	91.06		0.9802	0.18	2.3	
8.92	44.22	46.86		15.44	84.46	0.10		1.0886	2.82	6.60	90.58		0.9836	0.18	2.3	
10.40	42.89	46.71		17.88	82.03	0.09		1.1040	3.46	6.55	90.00		0.9875	0.19	2.4	
$CaCl_2$ = 0.05 in Dilute Phosphoric Acid																
1.47	49.03	46.84	2.66	2.70	91.87	0.10	5.33	1.0584	0.26	6.46	93.28		0.9731	0.10	1.4	
2.94	47.92	46.46	2.68	5.21	89.34	0.10	5.35	1.0738	0.67	6.41	92.92		0.9738	0.13	1.8	
4.45	46.75	46.11	2.69	7.63	86.89	0.10	5.37	1.0870	1.25	6.37	92.38	0.001	0.9760	0.16	2.2	1584
6.01	45.54	45.74	2.71	10.05	84.45	0.10	5.40	1.1008	1.93	6.32	91.74	0.001	0.9784	0.19	2.6	749
7.52	44.33	45.42	2.73	12.23	82.23	0.10	5.45	1.1157	2.80	6.28	90.91	0.002	0.9826	0.23	3.0	750
9.20	43.07	44.97	2.75	14.57	79.84	0.09	5.50	1.1300	3.83	6.25	89.92	0.002	0.9883	0.26	3.4	651
10.71	41.93	44.60	2.77	16.79	77.59	0.09	5.53	1.1441	4.61	6.22	89.16	0.003	0.9954	0.27	3.4	498
$CaCl_2$ = 0.10 in Dilute Phosphoric Acid																
1.50	47.19	45.89	5.41	2.61	86.68	0.10	10.61	1.1031	0.35	6.08	93.57	0.002	0.9732	0.13	1.9	729
3.02	45.97	45.57	5.44	5.01	84.24	0.10	10.66	1.1183	0.94	6.05	93.01	0.003	0.9748	0.19	2.6	654
4.56	44.86	45.09	5.49	7.21	81.96	0.10	10.73	1.1327	1.78	6.01	92.20	0.005	0.9777	0.25	3.4	562
6.08	43.69	44.70	5.53	9.38	79.72	0.10	10.81	1.1484	2.63	5.98	91.38	0.007	0.9815	0.28	3.7	455
7.60	42.51	44.32	5.57	11.43	77.58	0.09	10.90	1.1630	3.61	5.95	90.43	0.008	0.9859	0.32	4.1	443
9.38	41.09	43.93	5.61	13.69	75.19	0.09	11.03	1.1768	4.93	5.92	89.14	0.010	0.9939	0.36	4.6	398
10.76	39.99	43.61	5.64	15.53	73.26	0.08	11.13	1.1912	5.86	5.89	88.24	0.012	1.0047	0.38	4.7	352
$CaCl_2$ = 0.15 in Dilute Phosphoric Acid																
1.54	44.75	45.55	8.17	2.51	81.54	0.10	15.85	1.1518	0.51	5.73	93.75	0.018	0.9739	0.20	2.9	183
3.05	43.50	45.24	8.21	4.57	79.37	0.09	15.98	1.1653	1.45	5.70	92.82	0.034	0.9770	0.32	4.4	151
4.57	42.32	44.83	8.28	6.50	77.29	0.09	16.13	1.1804	2.55	5.68	91.72	0.049	0.9814	0.39	5.3	129
5.89	41.37	44.40	8.34	8.17	75.50	0.09	16.24	1.1948	3.51	5.65	90.77	0.069	0.9875	0.43	5.7	102
7.48	39.90	44.25	8.36	9.99	73.48	0.08	16.45	1.2089	4.93	5.63	89.33	0.113	0.9937	0.49	6.4	72
9.46	38.44	43.64	8.45	12.24	71.00	0.08	16.68	1.2235	6.66	5.61	87.58	0.159	1.0021	0.54	6.9	57
11.10	37.10	43.30	8.51	14.21	68.85	0.08	16.86	1.2358	8.02	5.59	86.18	0.213	1.0145	0.56	7.0	45
$CaCl_2$ = 0.20 in Dilute Phosphoric Acid																
1.55	42.59	44.83	11.03	2.28	76.56	0.09	21.06	1.1981	0.75	5.39	93.82	0.052	0.9755	0.33	4.6	133
3.05	41.33	44.52	11.10	4.06	74.59	0.09	21.26	1.2102	1.97	5.37	92.56	0.106	0.9823	0.48	6.7	97
4.65	40.07	44.10	11.18	5.75	72.64	0.09	21.52	1.2226	3.47	5.34	91.03	0.155	0.9892	0.60	8.2	84
6.23	38.81	43.70	11.26	7.49	70.69	0.08	21.74	1.2350	4.91	5.32	89.51	0.255	0.9969	0.66	8.7	56
7.83	37.49	43.35	11.33	9.23	68.73	0.08	21.96	1.2472	6.40	5.30	87.92	0.378	1.0052	0.69	9.0	40
9.41	36.17	43.03	11.39	11.14	66.60	0.08	22.18	1.2641	7.65	5.28	86.61	0.450	1.0144	0.69	8.7	34
11.06	34.84	42.62	11.48	13.18	64.45	0.08	22.30	1.2765	8.95	5.26	85.12	0.667	1.0246	0.68	8.3	23
$CaCl_2$ = 0.25 in Dilute Phosphoric Acid																
1.59	40.84	43.42	14.14	1.76	72.04	0.08	26.11	1.2450	1.40	5.07	93.11	0.423	0.9781	0.80	11.3	49
3.13	39.50	43.16	14.21	3.27	70.24	0.08	26.41	1.2594	2.98	5.05	91.44	0.537	0.9888	0.91	12.7	45
4.74	37.96	43.07	14.23	4.80	68.38	0.07	26.74	1.2738	4.68	5.03	89.60	0.695	1.0033	0.97	13.3	38
6.41	36.87	42.29	14.43	5.98	66.93	0.08	27.01	1.2884	6.86	5.01	87.04	1.092	1.0157	1.15	15.3	28
8.00	35.27	42.30	14.43	7.98	64.67	0.08	27.27	1.3015	8.02	4.99	85.79	1.190	1.0239	1.01	13.0	23
9.68	33.83	41.98	14.50	10.04	62.30	0.07	27.59	1.3148	9.32	4.97	84.46	1.245	1.0312	0.93	11.6	21

[a] w is the mass fraction; blanks: not exist or not detected.

indicator.[13] Calcium was assumed to transfer from the aqueous phase into the organic phase together with chloride ions.[6] The possibility of coextraction of hydrochloric acid into the organic phase can be neglected.[14] Water concentrations in aqueous phase and TBP concentrations in both phases can be determined by material balance.[15,16] All of the experiments are carried out at least two times, and the average values are taken as the results. The relative deviation is 0.2 % for phosphoric acid, 0.35 % for water, and 0.5 % for calcium chloride, respectively. The absolute deviation for volume, mass, and density is ± 0.015 mL, ± 0.0001 g, and ± 0.0001 g·mL^{-1}, respectively. The global experimental error is within 3 %.

Results and Discussion

All concentrations are determined in mass fraction w_i^{ϕ}, where ϕ stands for the phase (ϕ = o for the organic phase and ϕ = a for the aqueous phase) and i stands for the component (i = 1 for phosphoric acid, i = 2 for water, i = 3 for TBP, and i = 4 for calcium chloride). The composition of initial mixtures and equilibrium phases at 298.15 K are given in Table 1. The dilute phosphoric acid contains mass fractions of 0.028 to 0.20 phosphoric acid and 0.0 to 0.25 calcium chloride.

From Table 1, it can be seen that only a very small amount of TBP dissolves in the aqueous phase. Comparing the solubility of solvent in phosphoric acid/water/solvent systems with and without calcium chloride, it appears that TBP dissolves much less than butyl alcohol or isoamyl alcohol in the aqueous phase.[16−18] A considerable amount of water dissolves in the organic phase varying from 4.97 % to 6.87 %. The solubility of water in the organic phase decreases when the phosphoric acid concentration increases. This result is in agreement with previous studies on different systems and different phosphoric acid concentrations.[6−9] The solubility of water in the organic phase also decreases when the calcium chloride concentration

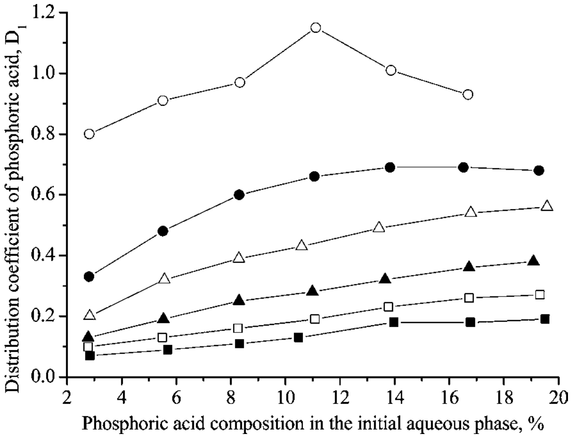

Figure 1. Distribution coefficient of phosphoric acid (D_1) as a function of phosphoric acid composition in the initial aqueous phase at 298.15 K: ■, $w = 0$ $CaCl_2$; □, $w = 0.05$ $CaCl_2$; ▲, $w = 0.10$ $CaCl_2$; △, $w = 0.15$ $CaCl_2$; ●, $w = 0.20$ $CaCl_2$; ○, $w = 0.25$ $CaCl_2$.

increases. This phenomenon has also been reported in the literature.[8,18,19] The solubility decrease of water can make a contribution to better phase separation which is also observed in our experiments. Besides, the solubility of calcium chloride in the organic phase increases gradually with increasing phosphoric acid and calcium chloride concentration.

To indicate the ability of TBP to recover phosphoric acid, distribution coefficients (D_i) of phosphoric acid (D_1) and separation factors (S_i) of water (S_2) and calcium chloride (S_4) are calculated and listed in Table 1.

The distribution coefficient D_i is defined as: $D_i = (w_i^o/w_i^a)$, and the separation factor S_i is defined as: $S_i = (D_1/D_i)$.

The variation of D_1, S_2, and S_4 as a function of phosphoric acid composition in the initial dilute phosphoric acid is shown in Figures 1 to 3.

In Figure 1, the results indicate that D_1 increases with increasing phosphoric acid concentration in the presence of $w = 0$ to 0.15 calcium chloride. When the concentration of calcium chloride is between $w = 0.20$ and 0.25, D_1 increases to a maximum value with increasing phosphoric acid concentration and then decreases with a further increase in the phosphoric acid concentration. The reason for the increase of D_1 is that phosphoric acid transfers into the organic phase only in a neutral form rather than a dissociated form.[20] As the phosphoric acid concentration increases, the proportion of undissociated phosphoric acid increases.[21] So phosphoric acid in the dilute phosphoric acid solution prefers to transfer into the organic phase. The decrease of D_1 may be attributed to the fixed amount of TBP, which can only extract a certain quantity of phosphoric acid. So a further increase in phosphoric acid concentration causes a decrease in the distribution coefficient of phosphoric acid.

The results also indicate that D_1 increases as the calcium chloride concentration increases. This phenomenon is due to the salt-out effect. Phase equilibrium is a result of balance of intermolecular forces, while adding calcium chloride can introduce ionic forces into the system and break the intermolecular balance. Since calcium ions and chloride ions are solvated by water molecules, the water molecules available for phosphoric acid are greatly reduced. Therefore, some phosphoric acid is salted out from aqueous solution into the organic phase.[22] So D_1 increases with increasing calcium chloride concentration. From Figure 1, it can also be concluded that TBP has a low capability to extract phosphoric acid from dilute phosphoric acid without calcium chloride, but TBP becomes an effective extractant for phosphoric acid as the calcium chloride concentration increases.

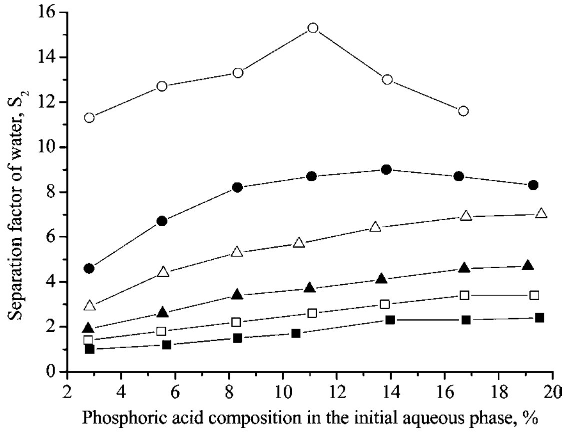

Figure 2. Separation factor of water (S_2) as a function of phosphoric acid composition in the initial aqueous phase at 298.15 K: ■, $w = 0$ $CaCl_2$; □, $w = 0.05$ $CaCl_2$; ▲, $w = 0.10$ $CaCl_2$; △, $w = 0.15$ $CaCl_2$; ●, $w = 0.20$ $CaCl_2$; ○, $w = 0.25$ $CaCl_2$.

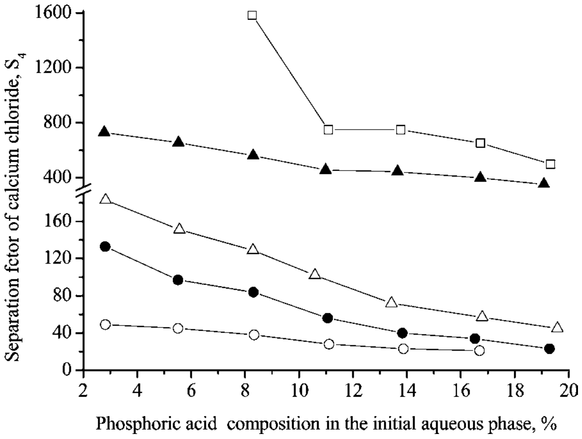

Figure 3. Separation factor of calcium chloride (S_4) as a function of phosphoric acid composition in the initial aqueous phase at 298.15 K: □, $w = 0.05$ $CaCl_2$; ▲, $w = 0.10$ $CaCl_2$; △, $w = 0.15$ $CaCl_2$; ●, $w = 0.20$ $CaCl_2$; ○, $w = 0.25$ $CaCl_2$.

In Figure 2, the results indicate that S_2 increases with an increase in phosphoric acid concentration in the presence of $w = 0$ to 0.15 calcium chloride; S_2 increases to a maximum value with an increase in phosphoric acid concentration and then decreases a little with a further increase in phosphoric acid concentration in presence of $w = 0.20$ to 0.25 calcium chloride. S_2 varies from 1.4 to 15.3 when the concentration of calcium chloride in dilute phosphoric acid is from $w = 0.05$ to 0.25.

In Figure 3, the results indicate that S_4 decreases with an increase in phosphoric acid and calcium chloride concentration. S_4 varies from 21 to 1584 when dilute phosphoric acid contains $w = 0.05$ to 0.25 calcium chloride.

On the basis of the results and discussion above, TBP is very selective since only small quantities of water and calcium chloride are present in the organic phase, which indicates the good ability of TBP to extract phosphoric acid from dilute phosphoric acid containing calcium chloride. TBP can be considered as a feasible and effective extractant for recovery of phosphoric acid from dilute phosphoric acid containing calcium chloride.

Conclusions

The liquid−liquid equilibrium data in the system phosphoric acid/water/TBP/calcium chloride has been determined at 298.15 K, in the concentration range of 0.028 to 0.20 mass fraction (w) phosphoric acid and 0 to 0.25 calcium chloride. The results show that TBP is very selective since only small quantities of water and calcium chloride are present in the organic phase, which indicates the good ability of TBP to extract phosphoric acid from dilute phosphoric acid containing calcium chloride. It can be concluded that TBP can serve as an adequate extractant to recover phosphoric acid from dilute phosphoric acid containing calcium chloride.

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