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Reactive Extraction of *o*-Aminophenol with Di(2-ethylhexyl)phosphoric Acid in Different Diluents

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Di(2-ethylhexyl)phosphoric acid was used as a reagent for the extraction of *o*-aminophenol in 2-octanol and kerosene. Equilibrium data for active and inactive diluents were obtained at various di(2-ethylhexyl)phosphoric acid concentrations. Both neutral and positive forms of *o*-aminophenol could be extracted by di(2-ethylhexyl)phosphoric acid. The maximum distribution coefficient occurred when the initial pH value was between pK_{a1} and pK_{a2}, the dissociation constants of *o*-aminophenol. At the same concentration the extractabilitiy of di(2-ethylhexyl)phosphoric acid increased as the polarity of the diluent increased. The difference of the distribution coefficient between polar diluents and nonpolar diluents decreased with an increase in the di(2-ethylhexyl)phosphoric acid concentration. Fourier transform infrared spectroscopic analysis was used to show that the behavior is controlled by ion association and ion exchange reactions.

Introduction

o-Aminophenol (OAP) has both a Lewis acid functional group (−OH) and a Lewis base functional group (−NH_2) attached to the benzene molecule, so it has specific physical and chemical characteristics. OAP is also an important chemical material, commonly used in the dye, pharmaceutical, and biological industries as an intermediate in the production of dyes and pharmaceuticals. It is a key component in industrial effluent from printing and dyeing mills, which is not biodegradable and has serious toxic and contaminative effects. There is no known feasible processing method for removing OAP from such effluent. Thus, it is necessary to adopt a reactive extraction method to recover the organic species from the effluent in order to reduce pollution.

Reactive extraction has high capacity and high selectivity to separate polar organic solutes from dilute solutions. It has received increasing attention for its simple methodology and low cost. Many organic acid and hydroxybenzene compounds have been studied, for example, succinate,[1,2] phenol,[3] catechol,[4] etc. Li et al.[5,6] and Wang et al.[7] have summarized the equilibrium rules of monocarboxylic acids extracted by trioctylamine (TOA) and studied their quantitative connection based on a correlative formula, which can guide the choice of a suitable extractant to separate monocarboxylic acids. To determine the range of use of a reactive extractant, researchers have studied the extraction of compounds that have two different functional groups. For example, Liu and co-workers[8−10] studied aminobenzoic acid, L-tryptophane, and L-isoleucine and suggested that there are both proton-transfer and ion-extraction reactions. Ihara et al.[11] proved that there was a better extraction of unprotected amino acids using mixed ligand nickel(II) and copper(II) chelates under neutral conditions. Li and co-workers[12,13] studied the extraction of *p*-aminophenol and established a mathematical model that describes the equilibrium behavior based on the physical extraction and reactive extraction of the solvent. Jin and Wang[14] investigated the removal of aminophenol in wastewater using di(2-ethylhexyl)phosphoric acid as the extractant. The experiments were conducted to study the effects of pH, oil/water ratio, and anti-extractant concentration. Qin et al.[15] conducted research on industrial effluents of processing dyestuff substrates, using TOA as the extractant and *n*-octanol and kerosene as the diluents. The research showed that the extraction efficiency depended on solution pH and aniline-2,5-disulfonic acid could be completely removed from the effluent using multistage cross-flow extraction and control of solution pH. In spite of good results and further advances, the research on amino acid species has been limited because of the complexities of the acids.

In this work, OAP was selected as the species to be extracted, di(2-ethylhexyl)phosphoric acid was studied for its ability to extract OAP, and *2*-octanol and kerosene were also investigated for their ability to affect the extraction power of the extractants over a wide pH range. IR spectroscopic analysis of the organic phase loaded with OAP was also investigated

Experimental Section

Chemicals. OAP from Fluka was AR grade with a purity of > 99 mass %. Di(2-ethylhexyl)phosphoric acid from a Beijing chemical reagent plant was CR grade with a purity > 97 mass %. The diluents (2-octanol, NaOH, and H_2SO_4) were from the Luoyang and Tianjin chemical reagent plant and were AR grade. All of the extractant and diluents were washed with distilled water to remove soluble impurities.

Kerosene was obtained from a local chemical plant. Fractional distillation was used for purification, and the kerosene was collected between (180 and 220) °C. The distilled kerosene was washed with concentrated sulfuric acid (98 %) ($V_{H_2SO_4}$:$V_{kerosene}$ = 1:5) twice and distilled water several times until the aqueous layer became neutral. The density of the sulfonated kerosene was about 0.78 g·cm^{-3}.

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Extraction Experiment. For the solution preparation, 0.1250 g of OAP was dissolved into a 0.5 L H_2SO_4 solution with a concentration of 0.05 mol·L^{-1}; di(2-ethylhexyl)phosphoric acid was used at a concentration of (0.2494, 0.5892, 1.495, and 2.991) mol·L^{-1} as the basis of the active and inactive diluents in the reactive extraction. Kerosene and 2-octanol were used as diluents, which can be classified as active and inactive, respectively.

OAP was extracted as follows: all extraction experiments were conducted with 100 mL flasks at 25 °C. Unless otherwise noted, 20 mL of the mixture solvents and 20 mL of the organic compound solution were added to each flask. The flask containing the mixture was shaken for about 1 h in a shaker bath with a vibrating rate of 200 rpm.

For clear separation of the two phases after each extraction, the mixed phases were centrifuged at 4000 rpm for 10 min or were left to equilibrate for 5 h or so. The upper layer (organic phase) was removed, and an aqueous phase sample was taken from the bottom layer (aqueous phase) for pH and solute concentration analysis.

Sample Analysis. After the pH value of the aqueous phase was measured with a pH meter (Hanna pH HI9321 model with a deviation of ± 0.01, calibrated with pH4.01/7.01/9.18 standard buffer solution, Italy), the pH of the aqueous phase was adjusted to 1−1.5 using concentrated H_2SO_4, and the aqueous samples were analyzed for the OAP concentration with a UV-250 spectrophotometer (Shimadzu, Japan) at 271 nm.

IR Spectra. The organic phase solutions were injected to a sample cell with a thickness of 0.05 mm between two CaF_2 plates. IR spectra were taken on a AVATAR360 FTIR spectrometer (Nicolet, America).

Results and Discussion

OAP has one Lewis acid group (−OH) and one Lewis base group (−NH_2). Two dissociation equilibria exist in aqueous solution as follows:

$$NH_3^+ArOH \overset{K_{a1}}{\rightleftharpoons} NH_3^+ArO^- + H^+ \tag{1}$$

$$NH_2ArOH \overset{K_{a2}}{\rightleftharpoons} NH_2ArO^- + H^+ \tag{2}$$

where pK_{a1} and pK_{a2} are 4.74 and 9.66, respectively.[16]

The cation form of OAP exists at pH < pK_{a1}, while the anionic form appears at pH > pK_{a2}, and neutral OAP dominates at intermediate pH. Therefore, the extraction equilibrium behavior of species with amphoteric functional groups are more complicated than those of non-amphoteric compounds. Therefore, it is essential to understand the effect of pH on the extraction.

Infrared Spectrogram of Reaction Extraction. Most studies of the acidic organophosphorus extraction of compounds with Lewis base functional groups have indicated the formation of an extraction complex with a neutral molecule or by ion exchange. However, other studies[8,9] have indicated that the extraction of an organic base by di(2-ethylhexyl)phosphoric acid forms an extraction complex by a proton-transfer reaction (formula 3) or an ion-exchange reaction (formula 4). In this work, all three forms of *o*-aminophenol exist but mainly in the form of H_2A^+ and HA. Ion-exchange and proton-transfer reactions were both demonstrated to exist by IR spectroscopic analysis of organic samples loaded with OAP in these extractions, without reference to active or inactive diluents. The reactive extraction of OAP with di(2-ethylhexyl)phosphoric acid can be described by the following reactions:

$$\overline{(HP)_2} + HA \rightleftharpoons \overline{HP{\cdot}P^-{\cdot}H_2^+A} \tag{3}$$

$$\overline{(HP)_2} + H_2A^+ \rightleftharpoons \overline{HP{\cdot}P^-{\cdot}H_2^+A} + H^+ \tag{4}$$

$$\overline{(HP)_2} \rightleftharpoons \overline{(HP)} + P^- + H^+ \tag{5}$$

where $(HP)_2$, H_2A^+, HA, and $P^-{\cdot}H_2^+A$ represent di(2-ethylhexyl)phosphoric acid, NH_3^+ArOH, NH_2ArOH, and the extraction complex, respectively.

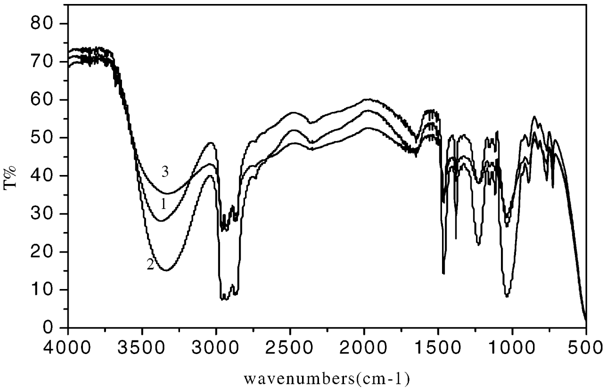

Figure 1. IR spectra for solutions containing di(2-ethylhexyl)phosphoric acid in 2-octanol (initial/equilibrium pH 1−2.97/2.78; 2−7.38/3.07; and 3−10.22/3.15).

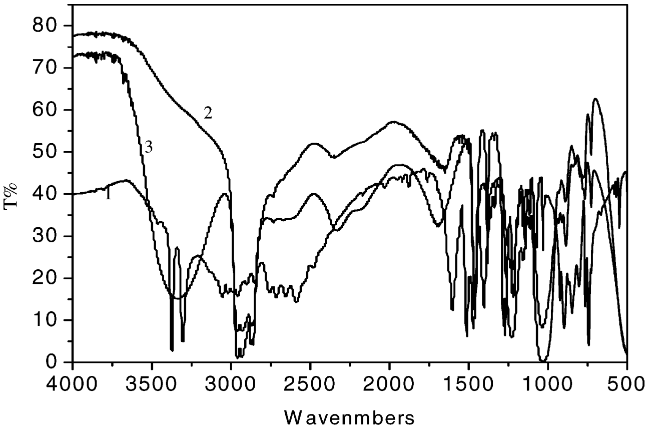

Figure 2. IR spectra of 1, *o*-aminophenol; 2, blank organic phase; 3, organic phase loaded *o*-aminophenol (initial/equilibrium pH 7.38/3.07).

As shown in Figure 1, the pH values have little effect on the structure of the complex, and compared with Figure 2, it is obvious that the peak at 1685.0 cm^{-1} assigned to the (P−OH) stretching vibration of di(2-ethylhexyl)phosphoric acid has been replaced by the peak at 1653.54 cm^{-1} assigned to −NH_3^+ unsymmetrical bending vibration. It is also obvious that the peak at 1604.50 cm^{-1} assigned to −NH_2 bending vibration was replaced by the peak at 1653.54 cm^{-1} assigned to −NH_3^+ unsymmetrical bending vibration. These analyses show that the extraction reactions are in agreement with either formula 3 or formula 4.

Figure 3 shows the IR spectrogram of normal heptane (because kerosene is a mixture) loaded with OAP. As can be seen, the structure of the reactive complex at different pH is independent of initial/equilibrium pH of the solution, and is similar to the result of Figure 1. The IR spectrogram showing kerosene loaded with OAP is in sharp contrast (Figure 4). The

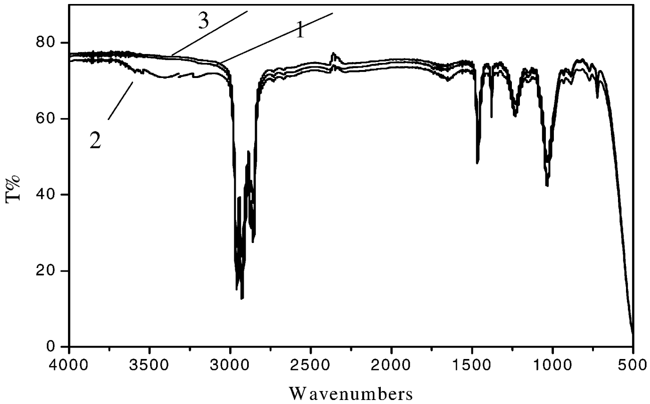

Figure 3. IR spectrogram for solution containing di(2-ethylhexyl)-phosphoric acid in normal heptane (initial/equilibrium pH 1−3.41/2.57; 2−7.35/2.94; and 3−10.26/3.36).

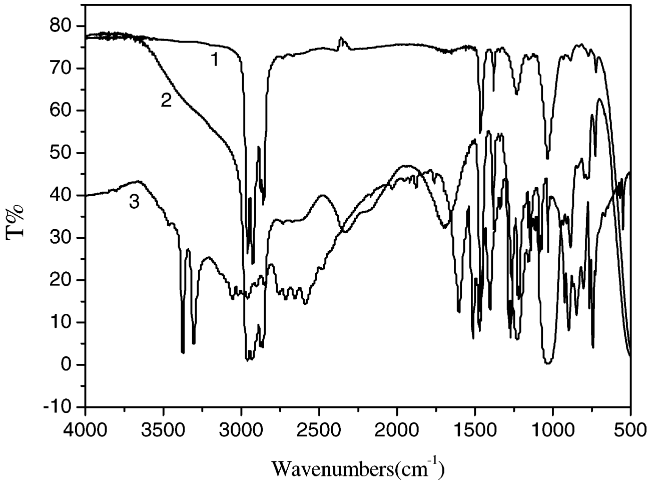

Figure 4. IR spectrogram of 1, organic phase loaded by *o*-aminophenol (initial/equilibrium pH 7.35/2.94); 2, blank organic phase; and 3, *o*-aminophenol.

Table 1. Equilibrium Data of *o*-Aminophenol between Water and Di(2-ethylexyl)phosphoric Acid in Kerosene

initial concn of extractant		total equilib concn of *o*-aminophenol/mg·L^{-1}		
mol·L^{-1}	initial/equilib pH in aq phase	aq phase	org phase	distribution coefficient
0.2494	3.12/2.68	147.1	102.9	0.69
0.2494	4.28/2.81	129.4	120.6	0.93
0.2494	5.45/2.87	119.8	130.2	1.09
0.2494	6.31/2.88	115.1	134.9	1.17
0.2494	7.07/2.9	113.8	136.2	1.19
0.2494	8.2/2.91	109.1	140.9	1.29
0.2494	8.91/2.92	112.8	137.2	1.22
0.2494	9.95/3.01	112.7	137.3	1.21
0.5892	3.26/2.62	69.7	180.3	2.59
0.5892	4.24/2.7	63.8	186.2	2.92
0.5892	5.37/2.73	61.2	188.8	3.08
0.5892	6.4/2.74	60.1	189.9	3.16
0.5892	7.17/2.75	57.4	192.6	3.36
0.5892	7.98/2.76	53.8	196.2	3.64
0.5892	8.88/2.77	59.5	190.5	3.20
0.5892	9.86/2.82	59.8	190.2	3.18
0.5892	10.74/2.83	69.1	180.9	2.62
0.8973	3.18/2.52	30.3	219.7	7.26
0.8973	4.32/2.57	28.3	221.7	7.82
0.8973	5.28/2.59	27.7	222.3	8.01
0.8973	6.28/2.61	25.5	224.5	8.81
0.8973	7.98/2.64	25.3	224.7	8.9
0.8973	8.97/2.65	29.4	220.6	7.51
0.8973	10.07/2.69	37.5	212.5	5.67
0.8973	11.1/2.74	40.1	209.9	5.23
1.4955	3.26/2.59	19.6	230.4	11.78
1.4955	4.62/2.63	19.1	230.9	12.08
1.4955	5.38/2.65	17.7	232.3	13.13
1.4955	6.54/2.66	17.6	232.4	13.23
1.4955	7.33/2.67	16.8	233.2	13.89
1.4955	8.16/2.68	18.3	231.7	12.68
1.4955	9.03/2.69	18.5	231.5	12.49
1.4955	10.11/2.71	19.8	230.2	11.66
1.4955	10.84/2.73	28.7	221.3	7.72
2.991	3.8/2.46	13.8	236.2	17.10
2.991	4.8/2.49	13.0	237.0	18.23
2.991	5.75/2.50	12.1	237.9	19.60
2.991	6.35/2.50	12.0	238.0	19.88
2.991	6.96/2.51	9.8	240.2	24.61
2.991	7.58/2.51	11.2	238.8	21.28
2.991	8.53/2.52	12.9	237.1	18.39
2.991	9.75/2.54	24.8	225.2	9.07
2.991	11.1/2.58	27.1	222.9	8.21

peak at 1685.0 cm^{-1} assigned to (P−OH) stretching vibration of di(2-ethylhexyl)phosphoric acid, the peak at 3600−3200 cm^{-1} assigned to $-NH_2$ stretching vibration, and the peak at 1604.50 cm^{-1} assigned to $-NH_2$ bending vibration of OAP all disappeared. The weak peak at about 1650 cm^{-1} assigned to $-NH_3^+$ unsymmetrical bending vibration appeared at the same time. It is also apparent that IR peak at 1229.18 cm^{-1} assigned to P═O of di(2-ethylhexyl)phosphoric acid has not moved, which demonstrates that ion-bonding and not hydrogen-bonding occurs in the reaction.

Effect of pH. The di(2-ethylhexyl)phosphoric acid molecule is a Lewis acid and, as such, can provide a proton. As such, it can react with the cationic and neutral forms of OAP to produce the complex that is extracted into the organic phase. Because the molar fractions of neutral and positive ions of OAP are a function of pH, the distribution coefficient (D) greatly depends on the pH of the aqueous phase. As is indicated by the data given in Tables 1 and 2, the distribution coefficient (*D*) increases with a sharp increase in equilibrium pH and then decreases at the same concentration of di(2-ethylhexyl)phosphoric acid. At the same time, it is apparent that the extraction of OAP by the di(2-ethylhexyl)phosphoric acid−2-octanol mixture is more affected than that of the di(2-ethylhexyl)phosphoric acid−kerosene mixture at a given pH.

The equilibrium solution pH is around 2.5−4 as shown in Tables 1 and 2, which is different from the initial solution pH (3.2−10) and increases with an increase in initial pH at the same concentration of di(2-ethylhexyl)phosphoric acid. Di(2-ethylhexyl)phosphoric acid is weak acid and has a pK_a of 3.32,[17] so it has a buffer capacity in the extraction process (see eq 5). As a result, the equilibrium solution pH remains between 2.5 and 4 as shown in Tables 1 and 2. This is different from the initial solution pH (3.2−10) and increases with an increase in initial pH at the same concentration of di(2-ethylhexyl)phosphoric acid.

The *D* change with respect to the equilibrium solution pH follows the same trend as that of the initial solution pH at the same concentration of di(2-ethylhexyl)phosphoric acid. However, the maximum *D* occurs at different equilibrium pH values, when the initial pH value is almost between pK_{a1} and pK_{a2}. The concentration of di(2-ethylhexyl)phosphoric acid is the controlling factor for the equilibrium pH value at which the maximum *D* occurs. Since the maximum *D* occurs when the initial pH value is between pK_{a1} and pK_{a2}, it indicates that the initial pH is also an important parameter on this extraction. The peak in *D* may be explained by the extraction mechanism given in the Infrared Spectrogram of Reaction Extraction section (i.e., all three forms of OAP exist in the pH region between pK_{a1} and pK_{a2}, but mainly as the neutral molecule), so OAP can react with di(2-ethylhexyl)phosphoric acid by ion-association and ion-

Table 2. Equilibrium Data of *o*-Aminophenol between Water and Di(2-ethylexyl)phosphoric Acid in 2-Octanol

initial concn of extractant		total equilib concn of *o*-aminophenol/mg·L^{-1}		
mol·L^{-1}	initial/equilib pH in aq phase	aq phase	org phase	distribution coefficient
0.2494	3.15/3.05	80.9	168.1	2.05
0.2494	4.13/3.25	57.1	192.9	3.38
0.2494	5.04/3.43	53.9	196.1	3.64
0.2494	6.09/3.44	42.1	207.9	4.39
0.2494	6.82/3.45	39.9	210.1	5.26
0.2494	7.24/3.46	39.4	210.6	5.34
0.2494	7.87/3.47	38.7	211.3	5.45
0.2494	8.95/3.49	37.3	212.7	5.67
0.2494	10.01/3.76	41.2	208.8	5.07
0.5892	2.99/2.88	63.1	186.9	2.96
0.5892	4.01/3.16	39.8	210.2	5.28
0.5892	5.12/3.21	38.4	211.6	5.51
0.5892	6.11/3.25	32.7	217.3	6.65
0.5892	6.86/3.26	32.6	217.4	6.67
0.5892	7.19/3.27	31.6	218.4	6.90
0.5892	8.2/3.3	31.2	218.8	7.02
0.5892	9.13/3.34	30.0	220.0	7.34
0.5892	10.13/3.43	33.1	216.9	6.56
0.8973	3.25/2.85	31.3	218.7	6.98
0.8973	4.6/2.96	25.8	224.2	8.70
0.8973	5.29/3.01	23.9	226.1	9.46
0.8973	6.28/3.04	23.1	226.9	9.84
0.8973	7.03/3.05	22.8	227.2	9.97
0.8973	7.98/3.07	21.1	228.9	10.88
0.8973	9.04/3.08	21.6	228.4	10.59
0.8973	10.12/3.13	26.0	224.0	8.61
0.8973	11.34/3.91	30.6	219.4	7.17
1.4955	3.73/2.76	15.0	235.0	15.67
1.4955	4.49/2.81	13.9	236.1	16.96
1.4955	6.62/2.88	12.6	237.4	18.81
1.4955	7.23/2.89	12.9	237.1	18.31
1.4955	7.83/2.90	14.0	236.0	16.83
1.4955	8.44/2.91	16.1	233.9	14.50
1.4955	9.71/2.96	19.4	230.6	11.91
1.4955	10.53/3.01	42.3	207.7	4.90
2.991	3.8/2.46	13.8	236.2	17.10
2.991	4.8/2.49	13.0	237.0	18.23
2.991	5.75/2.50	12.1	237.9	19.60
2.991	6.35/2.50	12.0	238.0	19.88
2.991	6.96/2.51	9.8	240.2	24.61
2.991	7.58/2.51	11.2	238.8	21.28
2.991	8.53/2.52	12.9	237.1	18.39
2.991	9.75/2.54	24.8	225.2	9.07
2.991	11.1/2.58	27.1	222.9	8.21

exchange reactions in which the ion-association reaction (see eq 3) plays a more important role.

Effect of Extractant Concentration. It can be seen from Figures 5 and 6 and Tables 1 and 2 that *D* increases when the extractant concentration increases, which likely results from the extraction equilibrium moving toward the direction of forming an extraction complex. It is also indicated in Figures 5 and 6 and Tables 1 and 2 that *D* is affected by the extractant concentration more greatly in the di(2-ethylhexyl)phosphoric acid−2-octanol system than in the di(2-ethylhexyl)phosphoric acid−kerosene system. The *D* value increased more obviously when the concentration increased from (0.2494 to 1.495) mol·L^{-1} than from (1.494 to 2.991) mol·L^{-1} in the di(2-ethylhexyl)phosphoric acid−2-octanol system. This mainly results from a descrease in the dissolving capacity of the extraction complex in this system with an increase in the extractant concentration, because the increase of the viscosity of di(2-ethylhexyl)phosphoric acid will hamper the dissolving capacity of the extraction complex.

Effect of Diluents. Tables 1 to 3 show the effect of the di(2-ethylhexyl)phosphoric acid concentration in various diluents on the distribution coefficient of OAP. As can be seen from Tables 1 and 2, the distribution coefficient value increased in the order 2-octanol > kerosene at the same di(2-ethylhexyl)phosphoric acid concentration and is similar to the order of the diluents polarity. The solubility of the reaction complex increased with the polarity of the diluents, so the reaction complex can exist more stably in the more active diluent.

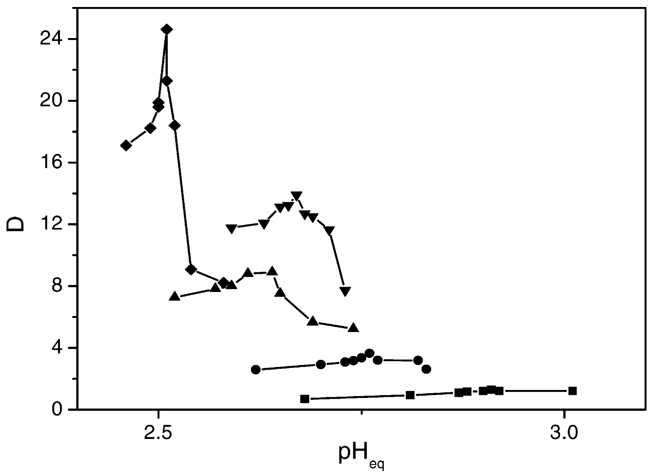

Figure 5. Extraction behavior of *o*-aminophenol with di(2-ethylhexyl)-phosphoric acid + kerosene. Di(2-ethylhexyl)phosphoric acid concentration: ■, 0.2494 mol·L^{-1}; ●, 0.5892 mol·L^{-1}; ▲, 0.8973 mol·L^{-1}; ▼, 1.4955 mol·L^{-1}; and ◆, 2.991 mol·L^{-1}.

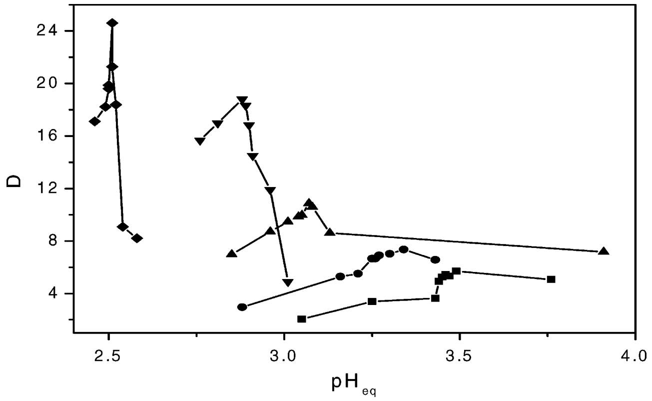

Figure 6. Extraction behavior of *o*-aminophenol with di(2-ethylhexyl)-phosphoric acid + 2-octanol. Di(2-ethylhexyl)phosphoric acid concentration: ■, 0.2494 mol·L^{-1}; ●, 0.5892 mol·L^{-1}; ▲, 0.8973 mol·L^{-1}; ▼, 1.4955 mol·L^{-1}; ◆, 2.991 mol·L^{-1}.

Table 3. Equilibrium Data (D_{max}) of *o*-Aminophenol in 2-Octanol and Kerosene

kind of diludents	D_{max}	pH
2-octanol	2.1205	6.83
kerosene	0.3380	7.27

It is also indicated in Figures 5 and 6 and Tables 1 and 2 that the difference of the *D* value between 2-octanol and kerosene decreased with an increase in the di(2-ethylhexyl)-phosphoric acid concentration (0 to 0.8973 mol·L^{-1}, except for 1.4955 mol·L^{-1}). There are probably three reasons for this result:

First, the physical extraction only by 2-octanol offers a high distribution coefficient (compared to kerosene, see Table 3), so its effect cannot be neglected. In addition, the presence of an active diluent will help dissolve the extraction complex. As a result, the difference of the *D* value between 2-octanol and kerosene is relatively large at the lower concentration of di(2-ethylhexyl)phosphoric acid.

Second, intermolecular hydrogen bonds exist between 2-octanol and di(2-ethylhexyl)phosphoric acid. Hydrogen bonding is stronger as the di(2-ethylhexyl)phosphoric acid concentration increases, which will make the formation of complex more difficult.

Third, the acidity of di(2-ethylhexyl)phosphoric acid allows the intramolecular hydrogen binding of OAP to become stronger, so as to make the reactive extraction more difficult. Therefore, the difference decreased more heavily at high concentration (0.8973 mol·L^{-1}) than at low concentration.

Conclusions

In this study, the extraction characteristics of OAP by di(2-ethylhexyl)phosphoric acid−kerosene and di(2-ethylhexyl)-phosphoric acid−2-octanol diluents were investigated. The extraction efficiency is higher when 2-octanol is added as diluent than that of kerosene at the same di(2-ethylhexyl)phosphoric acid concentration; The distribution coefficient increased with an increase in the di(2-ethylhexyl)phosphoric acid concentration; the maximum distribution coefficient value occurs when the initial pH value is between pK_{a1} and pK_{a2}. The infrared spectrogram demonstrated that this extraction reaction occurs via both ion-association and ion-exchange reactions, and the two reactions produced the same extraction complex.

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