Extraction of Glycolic Acid from Aqueous Solutions by Trioctyl Methylammonium Chloride and Organic Solvents

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The purpose of this study is to investigate the partitioning of glycolic acid from aqueous solutions by trioctyl methylammonium chloride, also known as Aliquat 336. Extraction equilibria of glycolic acid in the solution of trioctyl methylammonium chloride in mixtures of three individual diluting solvents and two solvent mixtures at a temperature of 298.15 K have been measured. The effect of initial acid concentration on the partitioning has been investigated. The solvents were propanol, cyclohexane, isooctane, butanol, toluene, methyl isobutyl ketone, and ethyl acetate. The distribution coefficients, *D*, loading factors, *Z*, and extraction efficiencies, *E*, are derived. The maximum removal of glycolic acid is 49% with propanol with a 1.76 mol·L^{-1} initial concentration of trioctyl methylammonium chloride.

Introduction

Carboxylic acids can be produced by the fermentation of biomass. They are promising intermediates because the oxygen of the biomass is placed in a form that is useful for further reaction with many other such products. The classical method for recovering nonvolatile hydroxycarboxylic acids, as glycolic acid from fermentation solutions, is the precipitation process. This method of recovery is costly and unfriendly to the environment.[1−3]

Because of their hydrophilic nature, hydroxycarboxylic acids are poorly extractable by common organic solvents; so, for their recovering from aqueous solutions, reactive liquid−liquid extraction has been considered. Aliphatic tertiary amines, with C_7 to C_{10}/C_{12} alkyl groups, have been proposed as suitable extractants for carboxylic and hydroxycarboxylic acids.[1−3] The amine extractants are dissolved in a diluent that dilutes the extractant to the desired concentration and controls the viscosity and density of the solvent phase. It has been found that, in the extraction of acids by amine, the type of diluent and the composition of the extraction mixture influence the equilibrium.

Polar diluents have been shown to be more convenient diluents than inert ones (nonpolar), due to the higher distributions.[4] However, active polar and proton-donating diluents such as alcohols have been shown to be the most suitable diluents for amines, because they give the highest distributions due to the formation of solvates through specific hydrogen bonding between the proton of the diluent and the acid−amine complex.[5,6]

Some authors suggest the formation of acid−amine complexes of only one type, (1,1) or (1,2), depending on the diluent, while others have suggested the formation of at least two.[1,5]

It has been found that diluents, especially those with functional groups, can affect the extraction behavior of amines significantly. The stoichiometry of the solute−amine complex, the loading of amine, as well as the third phase formation are influenced by the diluent. The effect of the diluent can be understood in terms of the ability to solvate to the organic phase species; therefore, it is necessary to distinguish between general solvation from electrostatic, dispersion, or other forces and specific solvation due to hydrogen bonding.[5]

The extraction process can be described by the reactions

$$\mathrm{HA} + \mathrm{R_4NCl} = \mathrm{HA{\cdot}R_4N} + \mathrm{Cl^-}$$

The resulting acid−amine complexes are supposed to be stabilizied due to the hydrogen bonding with the diluent.[2−7]

The structures of acid−amine complexes in diluents were determined by Barrow and Yerger.[8] They proposed that the first acid interacts directly with the amine to form an ion pair and the OH of the carboxyl of the second acid forms a hydrogen bond with the conjugated CO of the carboxylate of the first acid to form a complex.

Inci[9] and Qin et al.[10] have studied the extraction of glycolic acid by trioctylamine in different diluting solvents. They have found high distribution coefficients for glycolic acid.[9] However, data for glycolic acid, trioctyl methylammonium chloride, and diluent system have not been found in the literature. In the present work, the extraction of glycolic acid from aqueous solutions by trioctyl methylammonium chloride extractant in a variety of individual diluents and mixtures was examined in a wide range of amine concentrations (0.33 mol·L^{-1} to 1.76 mol·L^{-1}).

Extraction experiments were carried out with trioctyl methylammonium chloride dissolved in diluents of various types—ketone (MIBK), aromatic (toluene), different alkanes (isooctane and cyclohexane), different alcohols (propanol and butanol), and ester (ethyl acetate). As a result of batch extraction experiments, distribution coefficients were calculated. In addition to distribution coefficients, extraction efficiencies and loading factors were obtained. Furthermore, they were used to obtain conclusions about the stoichiometry of complex formation.

Theory

The extraction of glycolic acid (HA) with trioctyl methylammonium chloride (R_4NCl) can be described by the set of reactions

$$i\mathrm{HA} + j{*}\mathrm{R_4NCl} = {*}(\mathrm{HA})_i{\cdot}(\mathrm{R_4N})_j + j\mathrm{Cl^-} \qquad i = 1, p; j = 1, q \quad (1)$$

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where HA represents the nondissociated part of the acid present in the aqueous phase and organic phase species are marked with an asterisk. As no overloading of amine has been observed, i is expected to be less than or equal to j for any p and q. Reaction 1 can be characterized by the overall thermodynamic extraction efficiency, $^*K_{ij}$,

$$^*K_{ij} = {}^*(a_{\mathrm{HA}})_i\cdot(a_{\mathrm{R_4N}})_j{}^*(a_{\mathrm{Cl}^-})^j/(a_{\mathrm{HA}})^i{}^*(a_{\mathrm{R_4NCl}^-})^j \tag{2}$$

where a denote activities.

Equation 2 could be written in terms of dissociated species—hydrogen ions and acetate anions—as it is used in the literature on the amine extraction of acids.[10] Taking into account the dissociation equilibrium, one can derive that both concepts are equivalent, with the only difference being in the values of equilibrium constants. Replacing the activities by the products of molalities (moles per kilogram) and molal activity coefficients, γ, eq 2 takes the form

$$^*K_{ij} = {}^*(m_{ij}\gamma_{ij}){}^*(m_{\mathrm{cl}}\gamma_{\mathrm{cl}})^j/{}^*(m_{\mathrm{a}}\gamma_{\mathrm{a}})^i{}^*(m_{\mathrm{e}}\gamma_{\mathrm{e}})^j \tag{3}$$

where m_{a} is the molality of acid in the aqueous phase, m_{e} is the molality of amine in the aqueous phase, m_{e}° is the total molality of amine in the organic phase, m_{ij} is the molality of amine in the aqueous phase, γ_{a} is the molal activity coefficient of acid, γ_{e} is the molal activity coefficient of amine, and γ_{ij} is the molal activity coefficient of the complex.

The loading of the extractant, Z, is defined as the total concentration of acid in the organic phase divided by the total concentration of amine in the organic phase.[10] The expression for the loading, Z, can be writen in the form

$$Z = C_{\mathrm{a}}/C_{\mathrm{e,org}} \tag{4}$$

Distribution coefficients, D, for glycolic acid extracted from water into the organic phase were determined as

$$D = C_{\mathrm{a,org}}/C_{\mathrm{a}} \tag{5}$$

The efficiency of extraction, E, is expressed as

$$E = (1 - (C_{\mathrm{a}}/C_{\mathrm{a0}}))100 \tag{6}$$

where C_{a} is the concentration of acid in the aqueous phase after extraction and C_{a0} is the initial concentration of acid in the aqueous phase (moles per liter). An E value of 100% means that all of the acid in the aqueous phase has been removed and is present in the organic phase.[11]

Experimental Section

Trioctyl methylammonium chloride (Fluka, 91042, >99%), glycolic acid (Merck, 814186, solution 70% in water), propanol (Merck, 100995, >99.5%), cyclohexane (Merck, 2817, >99.7%), isooctane (Merck, 4727, >99.5%), *n*-butanol (Merck, 250220, >99%), toluene (Merck, 8325, >99.5%), methyl isobutyl ketone (Merck, 820820, >99%), and ethyl acetate (Merck, 822277, >99%) were used without purification.

Glycolic acid was dissolved in water to prepare the solutions with initial concentrations of acid of 8% (1.04 mol·L^{-1}). The initial organic phases were prepared by the dissolution of trioctyl methylammonium chloride in the diluents to produce solutions with approximately constant concentrations (1.76 mol·L^{-1}, 1.48 mol·L^{-1}, 1.17 mol·L^{-1}, 0.88 mol·L^{-1}, 0.59 mol·L^{-1}, and 0.33 mol·L^{-1}).

For distribution experiments, equal volumes of an aqueous glycolic acid solution and an organic solution of amine were stirred in glass flasks in a temperature controlled shaker bath at 25 °C for 2 h, which preliminary tests demonstrated to be a sufficient time for equilibration. Thereafter, the mixture was kept in a bath for another 6 h to 8 h to reach full separation of phases.

The concentration of the acid in the aqueous phase was determined by titration with aqueous 0.1 N sodium hydroxide (relative uncertainty 1%).[12] Acid analysis was checked against a material balance. In most cases, the deviation between the amount of acid analyzed and the amount of acid known from preparing the solutions by mass did not exceed 3%. Each of the experiments was repeated, and the relative uncertainties did not exceed 3%. The solubilities of amine salts and diluents in the aqueous phase were negligible in the range of variables investigated.

Results and Discussion

Table 1 presents results of the experimental investigation. The concentrations of trioctyl methylammonium chloride in solvents were between 0.33 mol·L^{-1} and 1.76 mol·L^{-1}. The glycolic acid concentration in the initial aqueous phase was 0.04 mol·L^{-1} (=8% w/w).

The equilibrium data on the distribution of glycolic acid between water and trioctyl methylammonium chloride dissolved in propanol, cyclohexane, isooctane, butanol, toluene, methyl isobutyl ketone, and ethyl acetate are presented in Table 1. It will be seen from Table 1 that a considerable amount of glycolic acid is removed by trioctyl methylammonium chloride from aqueous solutions. The amount of acid removed strongly depends on the trioctyl methylammonium chloride amount and the diluting solvent. The maximum removal of glycolic acid is 49% with propanol and a 1.76 mol·L^{-1} initial concentration of trioctyl methylammonium chloride. The acid concentration in water at equilibrium, C_{a}, decreases from 0.93 mol·L^{-1} to 0.28 mol·L^{-1} with an increase in the amount of trioctyl methylammonium chloride from 0.33 mol·L^{-1} to 0.76 mol·L^{-1} for ethyl acetate. The distribution coefficient increases from 0.42 to 0.95 with an increase in the amount of trioctyl methylammonium chloride from 0.33 mol·L^{-1} to 0.76 mol·L^{-1} for propanol. Isooctane has been found as the worst diluting solvent for the removal of glycolic acid (21.25%).

The extraction of carboxylic acids with tertiary amine–solvent systems can be explained by the formation of acid–amine complexes, which are affected by the diluents in different ways. Solvation of the complex by the diluent is a critical factor in the extraction of acid. The interactions between the complex and diluent can be divided into general solvation and specific interactions of the diluent with the complex.[12–15] Polar diluents have been shown to be more convenient diluents than inert ones (nonpolar), due to the higher distributions. However, in the extraction of glycolic acid by the quaternary amine used in this study, such an effect has not been found. It has been found that the polarity of the diluent is not important in the extraction of glycolic acid by a quaternary amine.[16]

Figure 1 shows the effect of organic solvents on the extraction of glycolic acid from aqueous solutions. It can be seen that the extraction power of the trioctyl methylammonium chloride–diluent mixture changes with increasing initial concentration of trioctyl methylammonium chloride in the organic phase.

In Figure 2, the effect of trioctyl methylammonium chloride concentration on loading is shown. The loading

Table 1. Molar Concentration of Amine in the Organic Phase, $C_{e,org}$, Molar Concentration of Acid in the Aqueous Phase, C_a, Molar Concentration of Acid in the Organic Phase $C_{a,org}$, Distribution Coefficient, *D*, Loading Factor, *Z*, and Extraction Efficency, *E*, for the Extraction of Glycolic Acid with Trioctyl Methylammonium Chloride and Individual Diluting Solvents

diluent	$C_{e,org}$ mol·L^{-1}	C_a mol·L^{-1}	$C_{a,org}$ mol·L^{-1}	*D*	*Z*	*E*
cyclohexane	0.33	1.01	0.020	0.020	0.063	2.00
	0.59	0.94	0.091	0.095	0.154	8.75
	0.88	0.90	0.132	0.146	0.150	12.75
	1.17	0.85	0.181	0.211	0.155	17.43
	1.48	0.80	0.232	0.287	0.156	22.31
	1.76	0.77	0.263	0.338	0.149	25.31
isooctane	0.33	0.99	0.040	0.040	0.124	3.93
	0.59	0.96	0.078	0.081	0.133	7.56
	0.88	0.92	0.118	0.129	0.135	11.43
	1.17	0.90	0.133	0.147	0.114	12.87
	1.48	0.84	0.192	0.226	0.13	18.50
	1.76	0.81	0.221	0.269	0.125	21.25
MIBK	0.33	0.74	0.296	0.398	0.898	12.62
	0.59	0.86	0.172	0.198	0.291	16.25
	0.88	0.90	0.137	0.151	0.155	20.37
	1.17	0.82	0.213	0.257	0.182	20.37
	1.48	0.82	0.215	0.261	0.145	20.75
	1.76	0.84	0.190	0.224	0.108	21.25
propanol	0.33	0.73	0.31	0.42	0.93	29.80
	0.59	0.69	0.34	0.49	0.58	33.31
	0.88	0.64	0.39	0.61	0.45	38.12
	1.17	0.62	0.41	0.65	0.35	39.50
	1.48	0.58	0.45	0.78	0.30	43.87
	1.76	0.53	0.50	0.95	0.28	48.81
butanol	0.33	0.88	0.158	0.179	0.480	15.25
	0.59	0.87	0.167	0.192	0.284	16.12
	0.88	0.85	0.182	0.213	0.207	17.56
	1.17	0.83	0.207	0.249	0.177	19.94
	1.48	0.81	0.223	0.273	0.151	21.50
	1.76	0.79	0.245	0.309	0.139	23.62
ethyl acetate	0.33	0.94	0.095	0.101	0.289	9.18
	0.59	0.91	0.121	0.132	0.206	11.68
	0.88	0.85	0.187	0.220	0.213	18.06
	1.17	0.82	0.214	0.259	0.183	20.62
	1.48	0.77	0.265	0.342	0.179	25.50
	1.76	0.74	0.295	0.396	0.167	28.37
toluene	0.33	1.03	0.0013	0.012	0.003	0.12
	0.59	0.97	0.068	0.070	0.116	6.62
	0.88	0.90	0.137	0.152	0.156	13.25
	1.17	0.86	0.18	0.21	0.17	17.31
	1.48	0.85	0.19	0.22	0.12	18.27
	1.76	0.83	0.21	0.25	0.11	20.19

curve is a plot of *Z* versus amine concentration. Overloading (loading greater than unity) indicates that complexes with more than one acid per amine have been formed. With all of the solvents, overloading cannot be observed.

For systems with only one amine per complex, there is no effect of total amine concentration on the loading. In this work, isooctane does not show any effect on the loading. If there is more than one amine per complex, loading increases with increasing amine concentration. The loading increases with increasing amine concentration for toluene and cyclohexane. Systems that exhibit aggregation, the formation of complexes with large numbers of acid and amine molecules, exhibit an abrupt increase in loading. In this work, the loading of other solvents (propanol, MIBK, butanol, and ethyl acetate) decreases, indicating that the complexes include the diluent and specifically complexes include more than one amine per complex.[17,18]

The equilibrium data on the distribution of glycolic acid between water and trioctyl methylammonium chloride dissolved in propanol−toluene, propanol−MIBK, and toluene−MIBK mixtures are presented in Table 2. Figure 3 shows the effect of diluting solvent mixtures on the extraction power of trioctyl methylammonium chloride. It can be seen that the extraction power of trioctyl methylammonium chloride is the strongest with the MIBK−propanol mixture. The maximum removal of glycolic acid is 33.18% with MIBK−propanol and a 1.76 mol·L^{-1} initial concentration of trioctyl methylammonium chloride. The acid concentration in water at equilibrium, C_a, decreases from 0.75 mol·L^{-1} to 0.69 mol·L^{-1} with an increase in the amount of trioctyl methylammonium chloride from 0.33 mol·L^{-1} to 0.76 mol·L^{-1} for MIBK−propanol. The distribution coefficient increases from 0.38 to 0.49 with an increase in the amount of trioctyl methylammonium

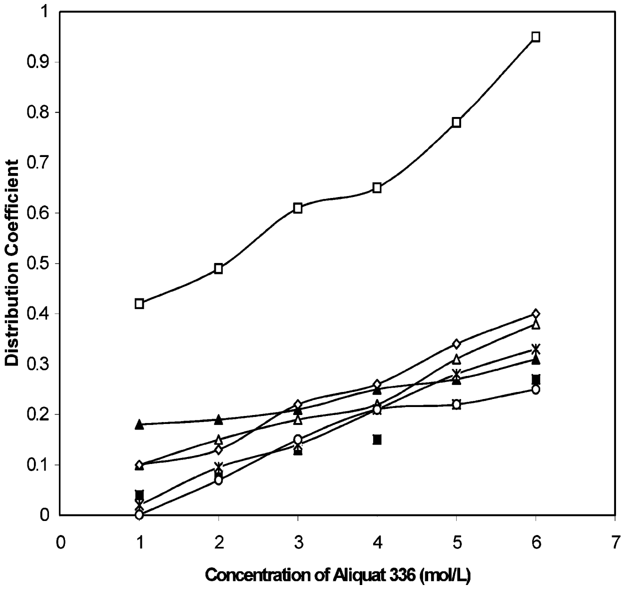

Figure 1. Distribution coefficients, *D*, vs the concentration of trioctyl methylammonium chloride in different individual diluting solvents: ■, cyclohexane; □, propanol; ▲, butanol; ◇, ethyl acetate; ○, toluene; ∗, isooctane; △, methyl isobutyl ketone.

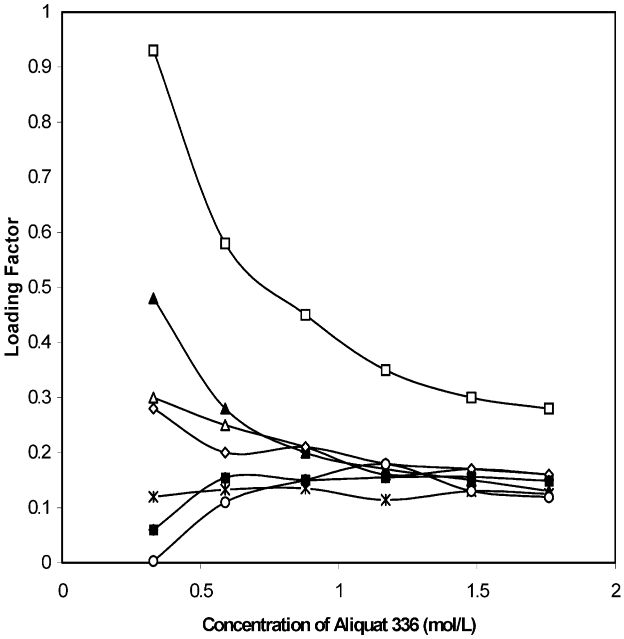

Figure 2. Loading factors, *Z*, vs the concentration of trioctyl methylammonium chloride in different individual diluting solvents: ■, cyclohexane; □, propanol; ▲, butanol; ◇, ethyl acetate; ○, toluene; ∗, isooctane; △, methyl isobutyl ketone.

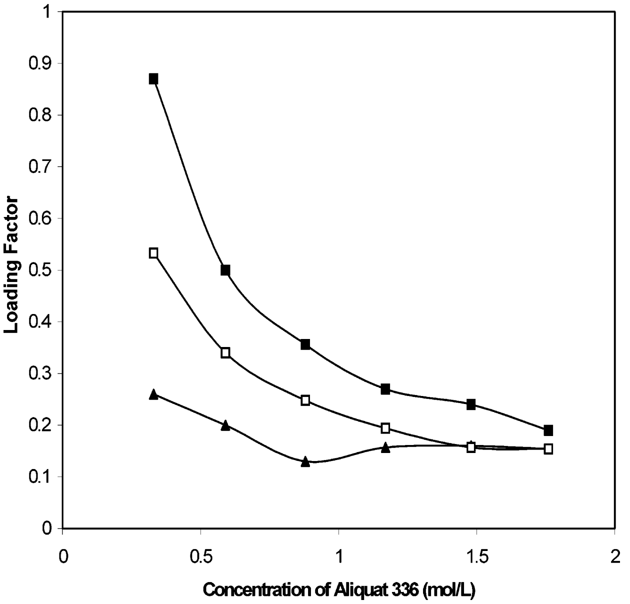

Figure 3. Distribution coefficients, *D*, vs the concentration of trioctyl methylammonium chloride in different solvent mixtures: ■, methyl isobutyl ketone + propanol; □, propanol + toluene; ▲, methyl isobutyl ketone + toluene.

Table 2. Molar Concentration of Amine in the Organic Phase, $C_{e,org}$, Molar Concentration of Acid in the Aqueous Phase, C_a, Molar Concentration of Acid in the Organic Phase, $C_{a,org}$, Distribution Coefficient, *D*, Loading Factor, *Z*, and Extraction Efficency, *E*, for the Extraction of Glycolic Acid with Trioctyl Methylammonium Chloride and Diluting Solvent Mixtures (50% + 50%)

diluent solvent mixture	$C_{e,org}$ mol·L^{-1}	C_a mol·L^{-1}	$C_{a,org}$ mol·L^{-1}	*D*	*Z*	*E*
MIBK + propanol	1.72	0.750	0.289	0.386	0.878	27.87
	0.92	0.741	0.298	0.402	0.505	28.68
	0.69	0.724	0.315	0.434	0.358	30.31
	0.69	0.718	0.321	0.447	0.275	30.93
	0.46	0.711	0.328	0.462	0.222	31.62
	0.23	0.694	0.345	0.496	0.196	33.18
MIBK + toluene	1.72	0.952	0.087	0.091	0.263	8.37
	0.92	0.921	0.118	0.128	0.200	11.37
	0.69	0.920	0.119	0.129	0.135	11.50
	0.46	0.855	0.184	0.215	0.157	17.75
	0.46	0.795	0.244	0.307	0.165	23.50
	0.23	0.768	0.271	0.353	0.154	26.12
propanol + toluene	1.72	0.863	0.176	0.203	0.533	16.93
	0.92	0.839	0.200	0.239	0.340	19.31
	0.69	0.821	0.218	0.265	0.248	21.00
	0.46	0.812	0.227	0.280	0.194	21.87
	0.46	0.806	0.233	0.289	0.157	22.43
	0.23	0.759	0.280	0.368	0.159	26.93

chloride from 0.33 mol·L^{-1} to 0.76 mol·L^{-1} for MIBK–propanol. The second best diluting solvent mixture for the removal of glycolic acid is propanol–toluene (20.93%), and the worst mixture used in this study is MIBK–toluene (21.12%). In Figure 4, the effect of solvent mixtures on the loading of trioctyl methylammonium chloride is presented.

Figure 5 shows the effect of the initial concentration of glycolic acid on the distribution coefficient. As the concentration increased, the distribution coefficient decreased. For example, the influence of the initial acid concentration on the glycolic acid distribution between water and trioctyl methylammonium chloride is demonstrated. It can be seen that the extraction power of trioctyl methylammonium chloride inreases with increasing initial concentration of glycolic acid in the aqueous phase.

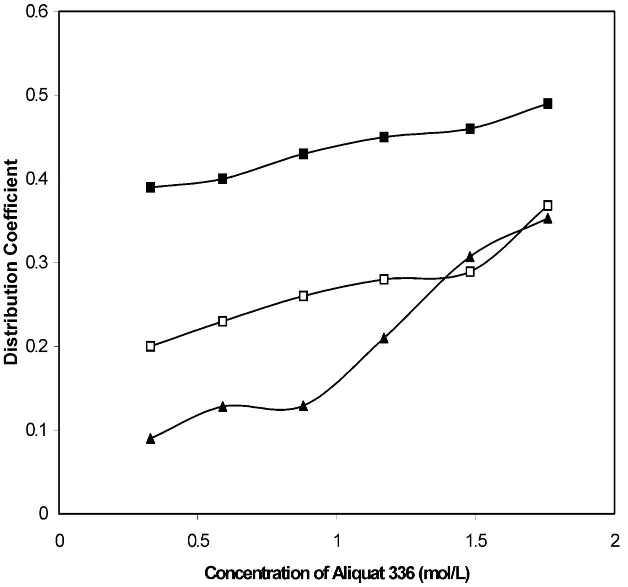

Figure 4. Loading factors, *Z*, vs the concentration of trioctyl methylammonium chloride in different solvent mixtures: ■, methyl isobutyl ketone + propanol; □, propanol + toluene; ▲, methyl isobutyl ketone + toluene.

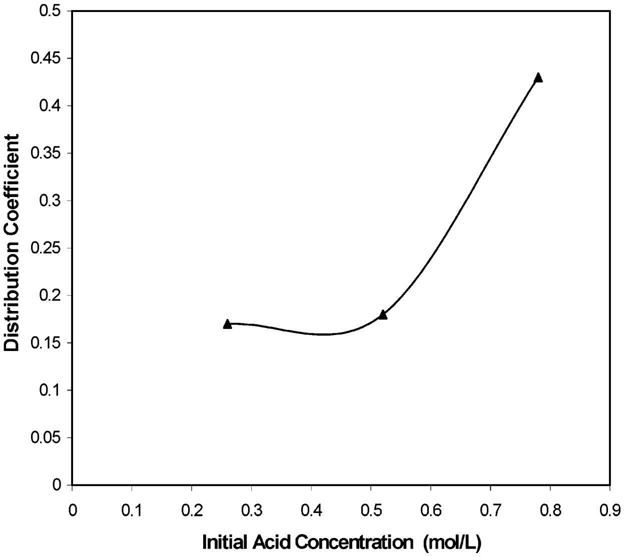

Figure 5. Distribution coefficients, *D*, for the extraction of glycolic acid with trioctyl methylammonium chloride at different intial acid concentrations.

Table 3. Distribution Coefficients, *D*, for Glycolic Acid between Solvents and Water

solvent	C_a mol·L^{-1}	$C_{a,org}$ mol·L^{-1}	*D*	*E* %
cyclohexane	0.99	0.05	0.05	4.81
isooctane	1.03	0.01	0.09	2.88
MIBK	1.01	0.03	0.03	2.89
propanol	0.67	0.36	0.54	35.31
butanol	0.92	0.12	0.13	11.54
ethyl acetate	1.00	0.04	0.04	3.84
toluene	0.99	0.05	0.05	4.81
MIBK + propanol	0.81	0.23	0.28	22.11
MIBK + toluene	0.94	0.1	0.11	9.61
propanol + toluene	0.88	0.16	0.18	15.38

The distribution data of glycolic acid between water and the solvents used in this study (propanol, cyclohexane, isooctane, butanol, toluene, methyl isobutyl ketone, and ethyl acetate) are presented in Table 3 and shown in Figure 6.

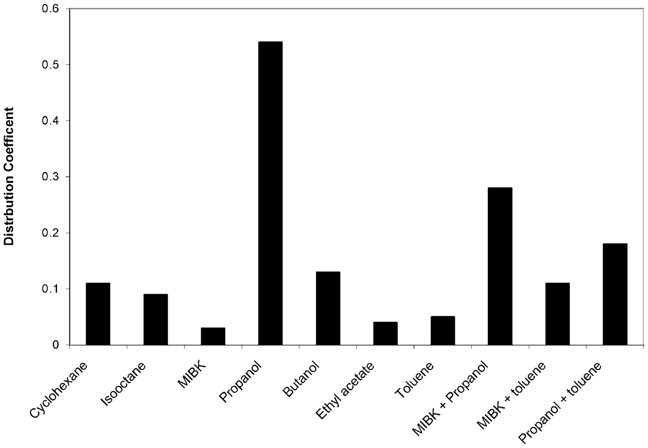

Figure 6. Distribution coefficients, *D*, for glycolic acid between water and the solvents used in this study.

Table 4. Molar Concentration of Amine in the Organic Phase, $C_{e,org}$, Molar Concentration of Acid in the Aqueous Phase, C_a, Molar Concentration of Acid in the Organic Phase, $C_{a,org}$, Distribution Coefficient, *D*, Loading Factor, *Z*, and Extraction Efficency, *E*, for the Extraction of Glycolic Acid with Trioctyl Methylammonium Chloride at Different Intial Acid Concentrations

initial acid concn	$C_{e,org}$	C_a	$C_{a,org}$			
mol·L^{-1}	mol·L^{-1}	mol·L^{-1}	mol·L^{-1}	*D*	*Z*	*E*
0.26	1.72	0.22	0.03	0.17	0.11	78.75
0.52	0.92	0.35	0.16	0.45	0.27	65.75
0.79	0.69	0.48	0.29	0.61	0.33	53.45

Conclusions

The aim of this study is to determine the extractability of glycolic acid from model aqueous solutions by trioctyl methylammonium chloride−diluent systems. Trioctyl methylammonium chloride is a compound consisting of an organic ion, Cl$^-$. This enables it to function essentially as an anion exchange reagent. In this study, the maximum removal of glycolic acid is 49% with propanol and a 1.76 mol·L^{-1} initial concentration of trioctyl methylammonium chloride. The maximum extraction efficiencies for diluents and their mixtures at maximum Aliquat are found to be the following: propanol > MIBK−propanol > ethyl acetate > MIBK > propanol−toluene > MIBK−toluene > cyclohexane > butanol > toluene > isooctane.

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