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Investigation of acrylic acid extractability from aqueous solution using tridodecyl amine extractant

Hasan Uslu

Engineering and Architecture Faculty, Chemical Engineering Department, Beykent University, Ayazağa, İstanbul, Turkey
Tel. +90 535 6220740; email: hasanuslu@gmail.com

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ABSTRACT

2-Propenoic acid (acrylic acid) extraction from aqueous solution using tridodecylamine (TDA) extractant dissolved in five single solvents (isoamyl alcohol, heptanol, hexanol, octanol, decanol) at 298K was studied. Some parameters related extraction, the extent to which the organic phase may be loaded with acrylic acid is expressed as loading ratio, Z, its values extraction efficiencies E and distribution coefficients, $K_{D,}$ were calculated in light of the experimental data. According to Bizek Approach, equilibrium complexation constants for (acid:amine) (1:1), (1:2) have been determined. The maximum removal of acrylic acid accomplished was about 91% with isoamyl alcohol having 1.188 mol.dm^{-3} initial concentration of TDA. Linear Solvation Energy Relationship (LSER) model was applied experimental data. LSER model results were compared with the experimental results and well agreement between them was observed.

Keywords: Reactive extraction; Acrylic acid; Tridodecylamine

1. Introduction

Acrylic acid is used primarily as an intermediate in the production of acrylates, which, in turn, are used in the production of polymers for coatings, paints, adhesives, paper and textiles. Acrylic acid is produced from the vapor phase oxidation of propylene or acrolein over an oxidation catalyst. The gaseous reactor effluent is then cooled and/or absorbed in water to obtain an aqueous solution containing from 10–80% acrylic acid and other impurities. The separation of acrylic acid from this aqueous stream has been difficult due to the relative volatilities of water [1].

Among the various alternatives methods (distillation, adsorption, electrodyalis etc.) for recovery of acrylic acid from the aqueous solution, solvent extraction appears to be the most promising one [2]. A principal disadvantage with recovery systems using solvents that have a boiling point lower than that of acrylic acid has been the need for a large amount of heat and solvent usage. An appropriate liquid-liquid extraction can provide high distribution coefficient, high selectivity and thus high economic potentials [3]. The improved results laid down the establishment of technology of reactive extraction for recovery of carboxylic acids. Organophosphorous compounds and secondary, tertiary and quaternary amine are widely employed to extract carboxylic acids. Out of the above extractants, tertiary amines have proved out to be most successful in recovery of carboxylic acids from dilute solutions [4–8]. An important study on the influence of diluents on amine extraction of carboxylic acids was carried out by Tamada and King [9,10].

Effect of extractant, diluent, initial acid concentration, extraction temperature are studied in literature [2–13]. Senol [10] investigated the equilibrium conditions for extraction of nicotinic acid between water and tri-*n*-octyl amine (TOA) dissolved in various diluents. Effect of diluents on the extractability, influence of acid structure and modelling of extraction system was discussed. Zhong et al. [11] studied the reactive extraction of propionic acid using Alamine 304-1 in 2-octanol, 1-dodecanol and

85 NF as diluents at various amine concentrations (0–100%) and found extraction to be maximum at the amine concentration between 20% and 40%.

The aim of this study was to explore the reactive extraction of acrylic acid from aqueous solutions using tertiary amine consisting of tridodecylamine (TDA) dissolved in several solvents. The extraction of acrylic acid from aqueous solutions by a TDA extractant in a variety of diluents was examined in a wide range of amine concentration (0.149–1.188 mol·dm^{-3}). Batch extraction experiments were performed with TDA dissolved in the diluents of alcohols (isoamyl alcohol, heptanol, hexanol, octanol and decanol). Distribution coefficients were calculated from the results of batch extraction experiments. In addition to distribution coefficients, complexation constants and variation of loading factors were calculated.

2. Theory

The extraction of acrylic acid (HA) with amine (R_3N) can be described by the reaction [14]

$$i\mathrm{HA} + j * \mathrm{R_3N} = * \left(\mathrm{HA}\right)_i \cdot \left(\mathrm{R_3N}\right)_j \quad i = 1, p, j = 1, q \tag{1}$$

where HA represents the nondissociated part of the acid present in the aqueous phase and organic phase species are marked with (*). As no overloading of amine has been observed i is expected to be smaller than or equal to j for any p and q. Reaction (1) can be characterized by the overall thermodynamic extraction constants;

$$\mathrm{K}_{i,j} = * \left[\left(\mathrm{HA}\right)_i \cdot \left(\mathrm{R_3N}\right)_j\right] / \left[\mathrm{HA}\right]^{i*} \left[\mathrm{R_3N}\right]^j \tag{2}$$

where square brackets denote activities.

It could be possible to study the extraction of acid by pure diluent in order to obtain the distribution coefficient but there is no evidence of the real value of this coefficient in the presence of amine and its complexes formed with the acid. The change of distribution coefficients with amine concentration can be caused by both the conditioned character of this constant and the stoichiometry of complex formation. Therefore, Loading factor must be taken into account consideration. The loading of the extractant, Z, is defined as the ratio of total concentration of acid in the organic phase, to the total concentration of amine in organic phase. The expression for the loading , Z, can be

$$Z = \frac{C_{\mathrm{org}}}{C_{\mathrm{e,org}}} \tag{3}$$

Distribution coefficients for acrylic acid extracted from water into organic phase were determined as,

$$K_D = \frac{C_{\mathrm{org}}}{C_{\mathrm{aq}}} \tag{4}$$

Degree of extraction is defined as the ratio of propionic acid concentration in organic phase to the sum of acid concentration in organic and aqueous phase and is defined in term of K_D as

$$E\% = \frac{K_D \mathrm{x} 100}{1 + K_D} \tag{5}$$

3. Experimental work

Acrylic acid, $C_3H_4O_2$, (M = 72.06 g·mol^{-1}) (purity > 99%) and TDA (tridodecylamine, Trilaurylamine), $C_{36}H_{75}N$, (M = 522.01 g·mol^{-1}) a commercial product (purity > 99%), were purchased from Merck Corp., USA. Other chemicals used without further purification in this study, are isoamyl alcohol (Merck > 99%), heptan-1-ol (Merck > 99), hexan-1-ol (Merck > %99), octan-1-ol (Merck > %99), and decan-1-ol (Merck > %99) were used without further purification.

Initial concentration of acrylic acid is 1.390 mol·dm^{-1} (10% in mass). The initial organic phases were prepared by the dissolving amine (TDA) in the diluents to produce solutions with approximately. Five different constant concentrations (1.118 mol·dm^{-3}, 0.981 mol·dm^{-3}, 0.594 mol·dm^{-3}, 0.297 mol·dm^{-3}, 0.149 mol·dm^{-3}) was prepared by combination of same volumes (10 ml) of aqueous and organic solutions in Erlenmeyer flasks. The solution putted to temperature controlled shaker (GFL) at 298K for 2 h which is found to be the sufficient time for reaching equilibrium during the preliminary tests. Then the mixture was kept in a bath for another 2 h to reach full separation of the phases.

The concentration of the acid in the aqueous phase was determined by titration with aqueous sodium hydroxide (relative uncertainty: 1%). Acid analysis was checked against a material balance. The volume of phases changing after the extraction was neglected so that the changing of the phase volume was very small. In most cases the deviation between the amount of acid analyzed and the amount of acid known by preparing the solutions by weighing did not exceed 3%. The acidity, pH of the aqueous phase measured with pH meter.

4. Results and discussion

4.1. Experimental results

Physical extraction of acrylic acid with pure diluents was investigated. Result of experiments of physical extraction was presented in Table 1. All diluent used in this study gave very poor extraction efficiency.

Table 1
Physical extraction of acrylic acid with pure diluents

Diluent	C_{aq} (mol·l^{-1})	C_{org} (mol·l^{-1})	K_D	E
Isoamyl alcohol	1.140	0.250	0.22	17.98
Hexanol	1.185	0.205	0.17	14.74
Heptanol	1.243	0.147	0.12	10.57
Octanol	1.281	0.109	0.08	7.84
Decanol	1.307	0.083	0.06	5.97

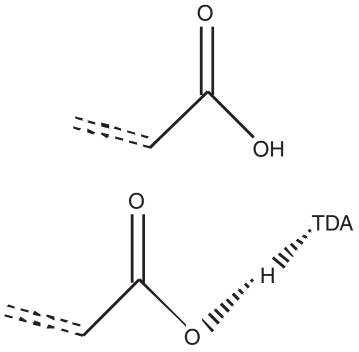

Fig. 1. Complex formation of acrylic acid and TDA.

The maximum distribution coefficient was reached by isoamyl alcohol as value of 0.22.

Table 2 presents results of the equilibrium data on the distribution of acrylic acid between water and TDA dissolved in isoamyl alcohol, heptan-1-ol, hexan-1-ol, octan-1-ol, and decan-1-ol. The concentrations of amines in diluents were between 0.149 mol·dm^{-3} and 1.118 mol·dm^{-3}. The initial concentration of acrylic acid was 1.390 mol·dm^{-3}.

Fig. 1 demonstrates the influence of the organic solvent on acrylic acid extraction between two phases. It can be seen that the extraction power of TDA - diluent mixture changes with increasing initial concentration of TDA in the organic phase. The most effective diluent was found as isoamyl alcohol for diluting TDA. It was found that the maximum removal of acrylic acid is 91.151% (wt) with isoamyl alcohol at 1.118 mol·dm^{-3} initial concentration of TDA. The aqueous phase acid concentration after the extraction, $C_{aq,}$ decreases from 1.030 mol·dm^{-3} to 0.123 mol·dm^{-3} with increasing amount of TDA from 0.149 mol·dm^{-3} to 1.118 mol·dm^{-3} for isoamyl alcohol. Distribution coefficient increases from 0.349 to 10.300 with the increase in the amount of TDA.

According to Table 1 and Fig. 2 the following obviously orders were found for TDA extraction.

Table 2
Results for extractions with individual diluting solvents

Diluent	$C_{e\,org}$ (mol·l^{-1})	C_{aq} (mol·l^{-1})	C_{org} (mol·l^{-1})	K_D	$K_{D,model}$	Z	E
Isoamyl alcohol	0.149	0.780	0.610	0.782	0.752	4.093	43.884
	0.297	0.539	0.851	1.578	1.546	2.865	61.223
	0.594	0.388	1.002	2.582	2.589	1.686	72.086
	0.891	0.281	1.109	3.946	3.620	1.244	79.784
	1.118	0.123	1.267	10.300	8.994	1.133	91.151
Hexanol	0.149	0.816	0.574	0.703	0.725	3.852	41.294
	0.297	0.635	0.755	1.188	1.326	2.542	54.316
	0.594	0.449	0.941	2.095	2.411	1.584	67.697
	0.891	0.306	1.084	3.542	3.683	1.216	77.985
	1.118	0.157	1.233	7.853	7.246	1.102	88.705
Heptanol	0.149	0.860	0.530	0.616	0.628	3.557	38.129
	0.297	0.674	0.716	1.062	0.948	2.410	51.510
	0.594	0.483	0.907	1.877	1.806	1.526	65.251
	0.891	0.338	1.052	3.112	3.337	1.180	75.683
	1.118	0.193	1.197	6.202	6.874	1.070	86.115
Octanol	0.149	0.918	0.472	0.514	0.620	3.167	33.956
	0.297	0.729	0.661	0.906	0.923	2.225	47.553
	0.594	0.560	0.830	1.482	1.561	1.397	59.712
	0.891	0.377	1.013	2.687	2.578	1.136	72.877
	1.118	0.225	1.165	5.177	5.730	1.042	83.812
Decanol	0.149	1.030	0.360	0.349	0.358	2.416	25.899
	0.297	0.846	0.544	0.643	0.624	1.831	39.136
	0.594	0.628	0.762	1.213	1.171	1.282	54.820
	0.891	0.414	0.976	2.357	2.542	1.095	70.215
	1.118	0.310	1.080	3.483	3.633	0.966	77.697

Isoamyl alcohol>> hexanol> heptanol> octanol> decanol

Polarity is the most effective factor to solve amine and formation of proper media to extract acid from aqueous solution. According to Carbon number of these solvents polarity is decreased with C number increased. This situation of high distribution coefficient can be explained by the formation of two or three acid: amine complexes, which are effected by the diluents in different way. In this study, using Bizek approach three acid: amine complexes, (Acid)(Amine); (Acid)(Amine)$_2$ and (Acid)$_2$(Amine)$_3$ have been assumed to exist in organic phase [9,14]. In this study, only K_{11} and K_{12} complexation constants have been calculated for alcohols according to Bizek's approach. 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. Inert diluents give a very low distribution of the acid into the solvent phase. Alkanes being nonpolar provide very low solvation of the polar complexes. Aromatic diluent give higher distribution, which has been rationalized as solvation due to interaction of the aromatic Π electrons with complex. Alcohols are polar and can promote extraction by providing a good dissolving media for the ion pair. However, polarity (or polarizability) alone does not completely account for solubility. Isoamyl alcohol diluent gives unusually high equilibrium constants, higher than would be expected from polarity arguments alone.

The values of the overall extraction constants, K_{11}, K_{12} are calculated (using eq. 2) and presented in Table 3.

According to Bizek approach [14], in the case of proton – donating diluent (alcohols) the extraction process can be described by the reactions;

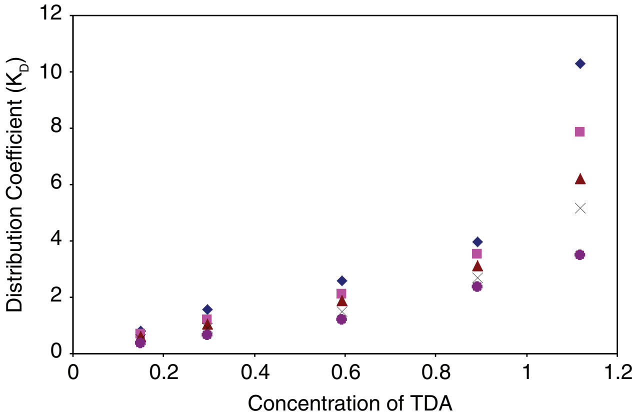

Fig. 2. Plot of distribution coefficients (K_D) against concentration of TDA in different individual diluting solvents: ♦, isoamyl alcohol; ■, hexanol; ▲, heptanol; X, octanol; •, decanol.

$$HA + {}^*R_3N = {}^*(HA)\cdot(R_3N) \qquad K_{11} \tag{6}$$

$$HA + 2\,{}^*R_3N = {}^*(HA)\cdot(R_3N)_2 \qquad K_{12} \tag{7}$$

The resulting acid:amine complexes are supposed to be stabilized due to hydrogen bonding with the diluent.

In the case of non-proton donating diluents (hexane, cyclohexane, toluene, MIBK) the process can be described the reaction;

$$2\,HA + 3{}^*R_3N = {}^*(HA)_2\,(R_3N)_3 \qquad K_{23} \tag{8}$$

Reaction (9) can be understood as a result of three consecutive reactions – (6), (7) and;

$$^*HA\,(R_3N) + {}^*(HA)(R_3N)_2 = {}^*(HA)_2\,(R_3N)_3 \quad K_A \tag{9}$$

The aggregation of highly polar primary acid: amine complexes according to reaction (8) are supposed to proceed almost completely. As the *(HA)(R$_3$N)$_2$ complex is minor in the studied range concentrations, its presence is not indicated.

Table 3
Results of overall extraction constants

Diluent	$C_{e\,org}$ (mol·l^{-1})	K_{11} (l. mol^{-1})	K_{21} (l^2. mol^{-1})
Isoamyl alcohol	0.149	1.325	1.699
	0.297	1.833	3.402
	0.594	2.230	5.747
	0.891	2.757	9.814
	1.118	5.950	48.380
Hexanol	0.149	1.192	1.461
	0.297	1.380	2.174
	0.594	1.809	4.030
	0.891	2.475	8.089
	1.118	4.536	28.897
Heptanol	0.149	1.044	1.214
	0.297	1.233	1.830
	0.594	1.621	3.357
	0.891	2.175	6.434
	1.118	3.582	18.564
Octanol	0.149	0.871	0.949
	0.297	1.053	1.444
	0.594	1.279	2.285
	0.891	1.877	4.980
	1.118	2.991	13.294
Decanol	0.149	0.592	0.575
	0.297	0.746	0.882
	0.594	1.047	1.668
	0.891	1.647	3.979
	1.118	2.012	6.492

The resulting acid-amine complexes are supposed to be stabilised due to the hydrogen bonding with the diluent [15,16]. The structure of acid amine complexes in diluents were determined by Barrow and Yerger [17]. 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 bonding with the conjugated CO of the carboxylate of the first acid to form a complex [18,19]. Fig. 1 shows the complex formation of acrylic acid and TDA.

The effect of concentration of TDA on loading is presented and shown in Fig. 3 and Table 1. The loading curve is a plot of *Z* versus amine concentration. Overloading have been observed at low amine concentration especially in between 0.149 and 0.297. For systems with only one amine per complex, there is no effect of total amine concentration on the loading. If there is more than one amine per complex loading increases with increasing amine concentration. Systems that exhibit aggregation, formation of complexes with large numbers of acid and amine molecules, causes increase in loading isoamyl alcohol and increase at 0.8–1.1 mol·dm^{-3} amine concentration indicates that complexes include large numbers of acid and amine molecules.

In Fig. 4, the concentration of TDA is varied from 0.149 mol·dm^{-3} to 1.118 mol·dm^{-3}. The initial concentration of acrylic acid is about 10%, w/w. As can be seen in Fig. 4, the increase in amine concentration causes gradual increase in extraction efficiency. Near 1.118 mol·l^{-1}, almost amount of 91% w/w acrylic acid is extracted with the help of isoamyl alcohol.

In Fig. 5, comparison of distribution coefficients of acrylic acid in pure individual solvents and TDA+diluents are presented. It was seen that the use of TDA dissolved in isoamyl alcohols increased the distribution coefficients about 47 times; dissolved in hexanol it increased about 46 times; dissolved in heptanol it increased about 52 times; dissolved in octanol it increased about 65 times; dissolved in decanol it increased about 58 times, all as compared to the use of pure solvents as extractant.

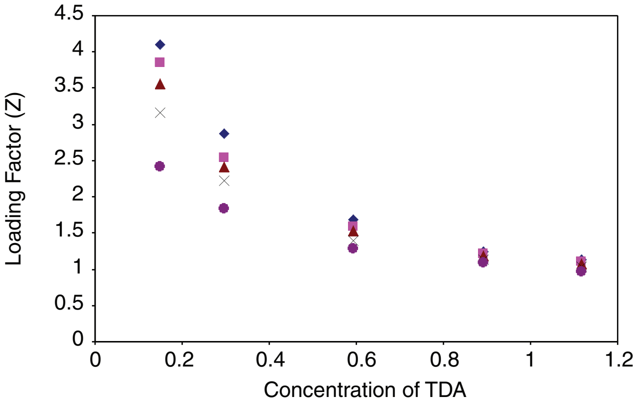

Fig. 3. Plot of loading factors (*Z*) against concentration of TDA in different individual diluting solvents: ♦, isoamyl alcohol; ■, hexanol; ▲, heptanol; X, octanol; •, decanol.

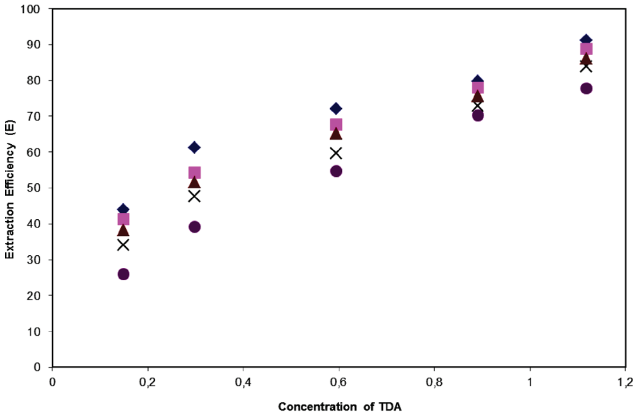

Fig. 4. Plot of l extraction efficiency (*E*) against concentration of TDA in different individual diluting solvents: ♦, isoamyl alcohol; ■, hexanol; ▲, heptanol; X, octanol; •, decanol.

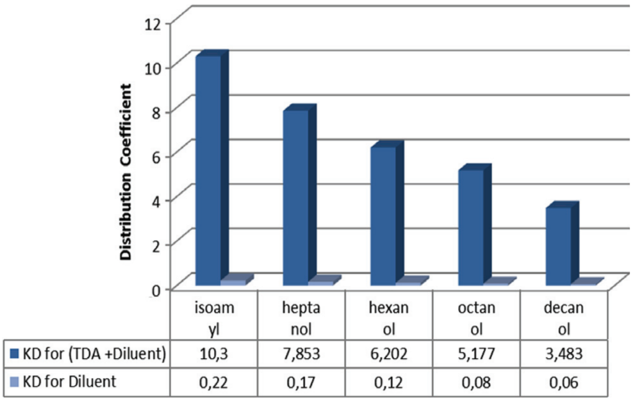

Fig. 5. Comparison of distribution coefficients of acrylic acid in pure individual solvents and TDA+solvents.

4.2. LSER model results

The properties of an acid/amine system of hydrogen bond formation can be estimated through theoretically based models of the mass action law including the physical interaction terms, or by using the concept of multiscale association, as well as by applying a generalized solvatochromic approach with linear solvation energy relationship (LSER) [20]. A modified version of LSER for predicting the extraction equilibria of an amine/diluent/acid system is given by Bizek et al. [14].

The calculations of distribution coefficients of the solvents in amine by LSER model equation were described in Uslu's earlier works [21,5]. Briefly, the following equation can be used to describe the effect of diluents on the values of distribution coefficients (K_D);

$$\ln K_D = \ln K_D{}^0 + s\left(\pi * + d\delta\right) + b\beta + a\alpha \tag{10}$$

The values of the distributions coefficients can be correlated according to equation 10 with the solvatochromic parameters of the solvents that come from the literature [22,23] shown in Table 4. The results are presented Table 1 as $K_{D,\mathrm{model}}$ and the calculated values show a good correlation to the experimental data. The estimated values of parameters of the model are presented in Table 5. It has been concluded that by using this model, distribution coefficients of acrylic acid between water and amine + diluent system can be described.

The root mean square deviations (RMSD) are calculated from the difference between the experimental data and the predictions of the LSER model according to the following equation:

$$\mathrm{RMSD} = \sqrt{\frac{1}{N}\sum_{i-1}^{n}\left(K_{D_{i,exp}} - K_{D_{i,calc}}\right)} \tag{11}$$

where $K_{Di,exp}$, is the experimental distribution coefficient, and $K_{Di,calc.}$ is the calculated distribution coefficient. N is the number of experimental data. The RMSD value of LSER model is found to be 0.089. The RMSD value shows all predicted distribution coefficients agree well with each other, and also the agreements between predictions and measurements are acceptable, taking into the consideration of the experimental uncertainty.

Table 4
Solvatochromic parameters for alcohols

Solvents	π^*	δ	β	α
Isoamyl alcohol	0.40	0	0.84	0.84
Hexan-1-ol	0.40	0	0.84	0.82
Heptan-1-ol	0.40	0	0.84	0.80
Octan-1-ol	0.40	0	0.81	0.77
Decan-1-ol	0.40	0	0.81	0.72

Table 5
The values of LSER model parameters *(D^0. s. d. b. a)* coefficient of linear regression. R^2. Standart Error. *SE*

LSER Model parameterss						
LnD^0	S	d	a	b	R^2	SE
1.572	−3.476	6.054	14.446	−3.482	0.925	0.300

5. Conclusion

The acrylic acid extraction from aqueous solution was investigated by TDA in different diluents. Isoamyl alcohol shows the highest extraction efficiency with about 91%. The extraction equilibrium was interpreted as a result of consecutive formation of two acid–amine species with stoichiometries of 1:1, 1:2. Overall thermodynamic extraction constants K_{11}, K_{12} have been determined. The results showed that the use of amines with polar diluent in the extraction is more efficient than physical extraction. Correlation of the results come from extraction by a linear solvation energy relationship-LSER model are suitable for the design of extractors.

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