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Extraction equilibria of propionic acid from aqueous solutions by Amberlite LA-2 in diluent solvents

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ABSTRACT

Amberlite LA-2 (a secondary amine) was studied for its ability to extract propionic acid at different amine concentrations. The extraction of propionic acid by Amberlite LA-2 dissolved in seven single solvents (cyclohexane, 2-octanone, toluene, methyl isobutyl ketone, isooctane, haxane and 1-octanol) was investigated under various amine concentrations at 298.15 K. Using Bizek's approach, two acids: amine *complexes*, (HO):(R_2N) and (HO):$(R_2N)_2$, are assumed to exist in the organic phase in case of proton-donating diluents, while the complexes (HO):(R_2N) and $(HO)_2$:$(R_2N)_3$ are suggested in the case of non-proton-donating diluents. Important data for design of separation units have been obtained as a result of batch experiments. These data are distribution coefficients (D), loading factors (Z), extraction efficiency (E) and overall extraction constants (K_{11}, K_{12}, K_{23}). The maximum removal of glycolic acid is 94.68% with MIBK and 0.93 mol L^{-1} initial concentration of Amberlite LA-2.

1. Introduction

Propionic acid is one of the most widely used carboxylic acid, which has many industrial applications [1]. It is an important carboxylic acid, finding ample uses ranging from animal feed preservation, including hay, silage and grains, human foods, baked goods to cheese. In addition to these major uses, propionic acid and derivatives have been used for manufacture of antiarthritic drugs, perfumes and flavors, plasticizers, mould preventives in silage and hay and as a solvent [2].

There are many patents on production of propionic acid, especially from petrochemical products [3]. However, new production processes of carboxylic acids have been investigated due to the high price of petrochemical products. Fermentation technology emerges as the best alternative providing the advantage of being energy efficient. Extractive fermentation technology provides the advantages of increased reactor productivity, produce, recovery of the fermentation product in one continuous step and hence reduction in downstream processing load and recovery cost [2,3].

Recovery of carboxylic acids from both aqueous solutions and fermentation broths, where it is present in dilute form (<10%), is always of interest to researchers [4]. Extensively, propionic acid is extracted from fermentation broth by calcium salt precipitation, which was found to be an expensive and an environmentally unfriendly technique. For the recovery of propionic acid by reactive extraction, common organic solvents such as ketones and alcohols show a low distribution ratio because of the high affinity of acid for water. Thus physical extraction with conventional solvents is not an efficient method for recovery of propionic acid. Reactive extraction employs a combination of extractant and diluent to enhance the recovery by intensifying the separation through simultaneous reaction and extraction and provides advantages of high selectivity and fractional recovery [5].

Amine compounds are useful and valuable extractans for separation of versatile cayboxylic acids which became the subject of last year's investigations. They are of importance because of their high efficiency and selectivity. This effect can provide much higher equilibrium distribution coefficients (KD) for extraction of carboxylic acids than solvents [6,7]. This improved result laid down the establishment of technology of reactive extraction for recovery of carboxylic acids. Organophosphorous compounds and secondary, tertiary, quaternary amines and their mixtures are widely employed to extract carboxylic acids [4,7].

Aliphatic secondary and tertiary amines, with C_7–C_{10}/C_{12} alkyl groups (TAA), have been proposed as suitable extractants for carboxylic and hydroxycarboxylic acids [8,9]. An organic solvent is required for several reasons, for example, to avoid the precipitation of the acid/amine complexes [10]. In addition, 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. Polar diluents have been shown to be more convenient diluents than inert ones (nonpolar), due to the higher distributions. However, active polar and proton-donating diluents as alcohols have been shown to be the most suitable diluents

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Nomenclature

Symbols

a_a	molal activity coefficient of acid
a_e	molal activity coefficient of amine
a_{ij}	molal activity coefficient of *complex*
$(c_a)^R$	concentration of acid in the aqueous phase (mol L^{-1})
$(c_a)^E$	concentration of acid in the organic phase (mol L^{-1})
$(c_e)^E$	concentration of amine in the organic phase (mol L^{-1})
K_A	aggregation constant
K_D	equilibrium distribution coefficient
K_{ij}	overall thermodynamic extraction constants
$(b_a)^R$	molality of acid in the aqueous phase (mol kg^{-1})
$(b_a)^E$	molality of acid in the organic phase (mol kg^{-1})
$(b_e)^R$	molality of amine in the aqueous phase (mol kg^{-1})
$(b_{eo})^E$	total molality of amine in organic phase (mol kg^{-1})
p	number of acid molecules
q	number of amine molecules
w	mass fraction
Z	loading factor
$(\,)^E$	organic phase
$(\,)^R$	aqueous phase

Abbreviations

D	distribution coefficient
HA	propionic acid
MIBK	methyl isobutyl ketone
R_2N	secondary amine

for amines, because they give the highest distributions resulting from the formation of solvates through specific hydrogen bonding between the proton of the diluent and the acid–amine complex [11–13].

Şenol has investigated extraction of differents carboxylic acids by reactive extraction [14–16]. Aljundi has investigated the removal of lactic acid by an adsorption column [17]. Wasewar et al. have investigated the removal of a lot of carboxylic acids by different extractants [2,4,18,19]. Also Mahfud has made a comprehensive investigation about extraction of some carboxylic acids [20,21]. Besides Uslu and Inci have studied the extraction of carboxylic acid with various materials [5,7]. However, no data is available about propionic acid and Amberlite LA-2/diluent system. The aim of present work is to investigate the extraction of propionic acid from aqueous solutions by Amberlite LA-2 with different diluent solvents in a wide range of amine concentration.

Extraction experiments were carried out with Amberlite LA-2 dissolved in the diluents of various types—ketone (MIBK, 2-octanone), different alkanes (isooctane, cyclohexane, hexane), alcohol (1-octanol), aromatic hydrocarbon (toluene). As a result of batch extraction experiments distribution coefficients were calculated. In addition to distribution coefficients, extraction efficiencies and loading factors have been obtained. Furthermore they were used to obtain conclusions about the stoichiometry of complex formation.

2. Theoretical

The extraction of propionic acid (HA) with amine (R_2N) can be described by the set of reactions:

$$i(\mathrm{HA})^R + j(\mathrm{R_2N})^E \rightleftarrows ((\mathrm{HA})_i\cdot(\mathrm{R_2N})_j)^E, \qquad i = 1,\ p; \quad j = 1,\ q \qquad (1)$$

where HA represents the undissociated part of the acid present in the raffinate or aqueous phase (^{R}) and extract (organic) phase species are marked with asterisk (^{E}). As no overloading of amine has been observed i is expected to be lesser than or equal to j for any p and q. Reactions (1) can be characterized by the overall thermodynamic extraction constants:

$$(K_{ij})^E = \frac{[(\mathrm{HA})_i\cdot(\mathrm{R_2N})_j]^E}{([\mathrm{HA}]^i)^R([\mathrm{R_2N}]^j)^E} \qquad (2)$$

where square brackets denote activities.

Eq. (1) could be written in terms of dissociated species—hydrogen ions and acetate anions as it is used in the literature on amine extraction of acids. Taking into account the dissociation equilibrium, one can derive that both concepts are equivalent, the only difference being in the values of equilibrium constants. Replacing the activities by the products of molalities (mol/kg of solvent) and molar activity coefficients, Eq. (2) takes the form:

$$(K_{ij})^E = \frac{(b_{ij})^E\cdot a_{ij}}{(b_a a_a)^{iE}(b_e a_e)^{jE}} \qquad (3)$$

where water and diluents are understood as solvents for the aqueous or organic phases, respectively.

As presented by Levien, the activity coefficients of undissociated propionic acid in water can be neglected in the first approximation [22,23]. Moreover, supposing the ratio of the activity coefficients of organic phase species being constant, it can be incorporated into the equilibrium constants. The conditional overall extraction constants are given by expressions:

$$K_{ij} = \frac{(b_{ij})^E}{(b_a^i)^R(b_e^j)^E}, \qquad i = 1, p; \ \ j = 1, q \qquad (4)$$

Combining Eq. (4) with the balance equations of acid and amine in the organic phase, the mathematical model of equilibrium is obtained in the form:

$$(b_a)^E = \sum\sum i K_{ij}(b_a^i)^R(b_e^j)^E, \qquad i = 1, p; \ \ j = 1, q \qquad (5)$$

where the molality of free amine is given by equation:

$$(b_e)^E + \sum\sum j K_{ij}(b_a^i)^R(b_e^j - b_e^0)^E = 0, \qquad i = 1, p; \ \ j = 1, q \qquad (6)$$

where b_e^0 is the total molality of amine in organic phase. Eq. (6) has a unique solution between zero and the aqueous phase molalities of undissociated acid, according to the dissociation equilibrium. As can be seen from the results by Vanura and Kuca and Sato et al., all possible (i,j) combinations for $i = 1, p$ and $j = 1, q$ need not to be taken into account [9,24]. 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 true value of this coefficient in the presence of amine and its complexes with the acid. The changes of K_{ij} with amine concentration can be caused by both the conditional character of this constant and the stoichiometry of complex formation. 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 organic phase. The expression for the loading, Z, can be derived from Eqs. (5) and (6) in the form:

$$Z = \frac{(b_a)^E}{(b_e^0)^E} \rightleftarrows \frac{\sum iK_{i1}(b_a)^R}{1+\sum K_{i1}(b_a^i)^R} \qquad i = 1, p; \ \ j = 1, q \qquad (7)$$

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

$$D = \frac{(b_a)^E}{(b_a)^R} \qquad (8)$$

3. Experimental

Amberlite LA-2, a commercial product (Henkel Co.) was used—a mixture of straight-chain secondary amine mixture ($M = 374$ g/mol). Propionic acid (Merck, >99%), hexane (Merck, >99%), cyclohexane (Merck, >99%), toluene (Carlo Erba, >99%), isooctane (Merck, >99%), MIBK (Merck, >99%), 2-octanone (Merck, >99%) and 1-octanol (Merck, >99%) were used without further purification.

The appropriate amounts of propionic acid were dissolved in water to prepare the solutions with initial concentrations of acid of 1.61 mol L^{-1} ($w = 10.65\%$). The initial organic phases were prepared by the dissolution of amine in the diluents to produce solutions with approximately constant concentrations (0.93 mol L^{-1}, 0.74 mol L^{-1}, 0.56 mol L^{-1}, 0.37 mol L^{-1}, and 0.19 mol L^{-1}). Known volumes of aqueous and organic solutions of known concentrations were added to Erlenmeyer flasks and equilibrated in a temperature controlled shaker bath at 298.15 K for 2 h. This was found to be a sufficient time for equilibration, as determined by preliminary testing procedures. Thereafter, the mixture was kept in a bath for another 6–8 h to reach full separation of phases.

The concentration of the acid in the aqueous phase was determined by titration with aqueous sodium hydroxide (relative uncertainty: 1%) [22]. 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 by preparing the solutions by weighing did not exceed 3%. The solubilities of amine salts and diluents in the aqueous phase were negligible in the range of variables investigated.

4. Results and discussion

Table 1 presents results of the experimental investigation. The concentrations of amines in solvents were between 0.19 mol L^{-1} and 0.93 mol L^{-1}. The propionic acid concentration in the initial aqueous phase was 1.61 mol L^{-1} ($w = 10.65\%$).

The equilibrium data on the distribution of propionic acid between water and Amberlite LA-2 dissolved in hexane, cyclohexane, toluene, methyl isobutyl ketone, isooctane, 2-octanone and 1-octanol are presented in Table 2. The values of distribution coefficients were found to follow the trend 1-octanol > 2-octanone > MIBK > isooctane > toluene > cyclohexane > hexane. Propionic acid has a high affinity to water and low relative volatility that renders it difficult to separate. The low activity of propionic acid toward these diluents, i.e., its higher solubility in water than in organic solvents, is the cause of the low distribution coefficient. The conventional extraction techniques are thus unprofitable. Better possibilities are offered by the reactive extraction technique which have proved to be effective in the recovery of carboxylic acids [25].

Fig. 1 demonstrates the influence of the organic solvent on propionic acid distribution between water and Amberlite LA-2. It can be seen that the extraction power of Amberlite LA-2–diluent mix-

Table 1
Experimental results of the extraction of propionic acid with Amberlite LA-2/individual diluting solvents.

Diluent	$(C_e)^E$ (mol L^{-1})	$(C_a)^R$ (mol L^{-1})	$(C_a)^E$ (mol L^{-1})	D	Z	E (%)
Cyclohexane	0.19	0.84	0.77	0.92	4.17	47.97
	0.37	0.56	1.05	1.88	2.84	65.32
	0.56	0.52	1.09	2.09	1.98	67.68
	0.74	0.27	1.34	4.90	1.81	83.053
	0.93	0.24	1.37	5.62	1.48	84.88
Isooctane	0.19	0.84	0.77	0.92	4.16	47.78
	0.37	0.50	1.11	2.23	3.00	69.03
	0.56	0.32	1.29	4.09	2.35	80.34
	0.74	0.19	1.42	7.28	1.91	87.92
	0.93	0.15	1.46	9.43	1.57	90.41
MIBK	0.19	0.36	1.25	3.43	6.73	77.43
	0.37	0.25	1.36	5.43	3.67	84.45
	0.56	0.18	1.43	7.84	2.60	88.69
	0.74	0.12	1.49	12.74	2.02	92.72
	0.93	0.09	1.52	17.80	1.65	94.68
1-Octanol	0.19	0.29	1.32	4.58	7.14	82.09
	0.37	0.28	1.33	4.78	3.60	82.70
	0.56	0.22	1.39	6.47	2.53	86.62
	0.74	0.13	1.48	11.70	2.00	92.12
	0.93	0.10	1.51	15.91	1.64	94.09
2-Octanone	0.19	0.30	1.32	4.50	7.12	81.83
	0.37	0.28	1.33	4.70	3.60	82.70
	0.56	0.17	1.44	8.27	2.61	89.21
	0.74	0.12	1.49	12.27	2.01	92.47
	0.93	0.09	1.52	16.41	1.64	94.26
Toluene	0.19	0.66	0.95	1.44	5.14	59.09
	0.37	0.37	1.24	3.38	3.36	77.16
	0.56	0.21	1.40	6.52	2.54	86.71
	0.74	0.14	1.47	10.92	1.99	91.61
	0.93	0.11	1.50	14.32	1.63	93.47
Hexane	0.19	0.85	0.76	0.89	4.11	47.21
	0.37	0.49	1.12	2.27	3.02	69.39
	0.56	0.33	1.28	3.89	2.33	79.55
	0.74	0.22	1.39	6.47	1.88	86.62
	0.93	0.16	1.45	9.34	1.57	90.33

Table 2
The values of the overall extraction constants.

Diluent	$(C_e)^E$ (mol L^{-1})	K_{11} (L mol^{-1})	K_{23} (L^4 mol^{-4})	K_{12} (L^2 mol^{-2})
Cyclohexane	0.19	4.98	173.89	–
	0.37	5.09	66.62	–
	0.56	3.74	22.92	–
	0.74	6.62	44.35	–
	0.93	6.04	28.70	–
Isooctane	0.19	4.94	171.95	–
	0.37	6.02	88.31	–
	0.56	7.30	73.54	–
	0.74	9.83	92.33	–
	0.93	10.14	75.98	–
MIBK	0.19	18.54	1491.68	–
	0.37	14.68	428.40	–
	0.56	14.01	245.44	–
	0.74	17.22	268.50	–
	0.93	19.14	258.59	–
1-Octanol	0.19	24.78	–	133.94
	0.37	12.92	–	34.93
	0.56	11.56	–	20.64
	0.74	15.81	–	21.36
	0.93	17.11	–	18.40
2-Octanone	0.37	24.34	2432.57	–
	0.37	12.92	339.20	–
	0.56	14.76	271.08	–
	0.74	16.59	249.86	–
	0.93	17.65	220.74	–
Toluene	0.19	7.81	346.47	–
	0.37	9.13	181.53	–
	0.56	11.65	173.59	–
	0.74	14.76	199.67	–
	0.93	15.40	169.52	–
Hexane	0.19	4.83	166.27	–
	0.37	6.13	90.88	–
	0.56	6.95	67.29	–
	0.74	8.75	74.19	–
	0.93	10.04	74.57	–

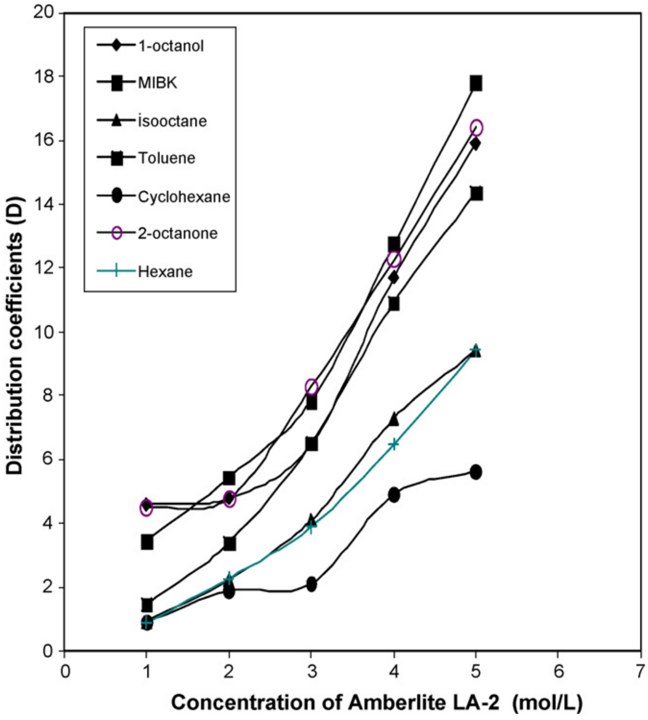

Fig. 1. Variation of distribution coefficients with concentration of Amberlite LA-2 in different individual diluting solvents.

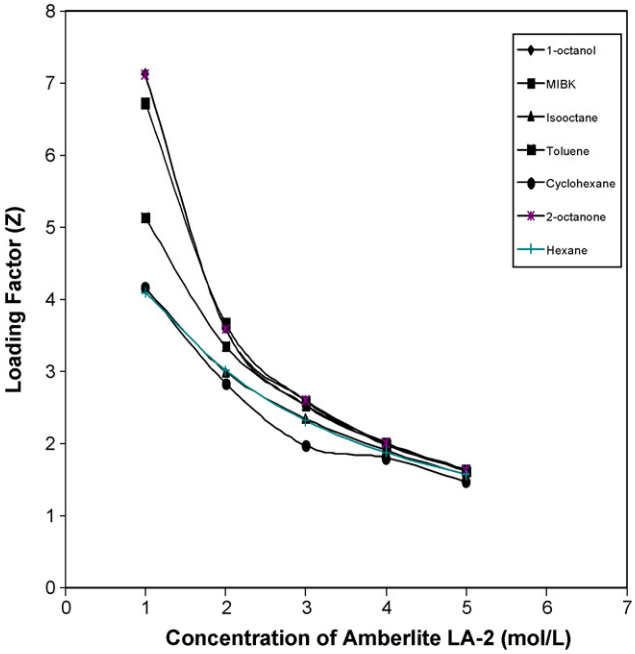

Fig. 2. Variation of loading factors with concentration of Amberlite LA-2 in different diluting solvents.

ture changes with increasing the initial concentration of Amberlite LA-2 in the organic phase.

According to Table 1 and Fig. 1 for Amberlite LA-2 extraction, the following orders were found for the respective.

MIBK > 2-octanone > 1-octanol > toluene > isooctane > hexane > cyclohexane.

This fact can be explained by the formation of two or three acids: amine complexes, which are effected by the diluents in different ways. In this study, using Bizek's approach three acids: amine *complexes*, $(HO)\cdot(R_2N)$; $(HO)\cdot(R_2N)_2$ and $(HO)_2\cdot(R_2N)_3$ have been assumed to exist in organic phase [16,22].

Solvation of the complex by the diluent is a critical factor in the extraction of acid. The interactions between the *complex* and the diluent can be divided into the general solvation interactions and specific interactions of the diluent with the *complex*. Inert diluents, such as non-polar alkanes hexane, isooctane and cyclohexane, provide very low solvation of the polar complexes. Thus they do not contribute significantly to the distribution of the acid into the solvent phase, and give low values of distribution coefficients. Aromatic diluent (toluene) gives higher distribution coefficient, which has been rationalized in terms of solvation due to the interaction of the aromatic π-electrons with the complex. MIBK and 2-octanone are polar and can promote extraction by providing a good media for the ion pair. However, polarity (or polarizability) alone does not completely account for the solvating ability. Capability of hydrogen bonding is important in case of using alcohol diluent as a result of this effect and 1-octanol has shown high distribution coefficient in this study [22].

In Fig. 2, the effect of Amberlite LA-2 concentration on loading is shown. The loading curve is a plot of Z vs. amine concentration. Overloading (loading greater than unity) indicates that complexes with more than one acid per amine have been formed. Overloading can be observed with all of the solvents, especially at high Amberlite LA-2 concentrations (Fig. 2).

For systems with only one amine per *complex*, there is no effect of total amine concentration on the loading. If there is more than

Table 3
Distribution of propionic acid between solvents and water.

Diluent	$(C_a)^R$ (mol L^{-1})	$(C_a)^E$ (mol L^{-1})	D	E (%)
Cyclohexane	1.408	0.15	0.11	9.71
Isooctane	0.901	0.66	0.73	42.23
MIBK	0.689	0.87	1.26	55.80
1-Octanol	0.521	1.04	1.99	66.59
2-Octanone	0.612	0.95	1.55	60.72
Toluene	1.088	0.47	0.43	30.23
Hexane	1.417	0.14	0.10	9.09

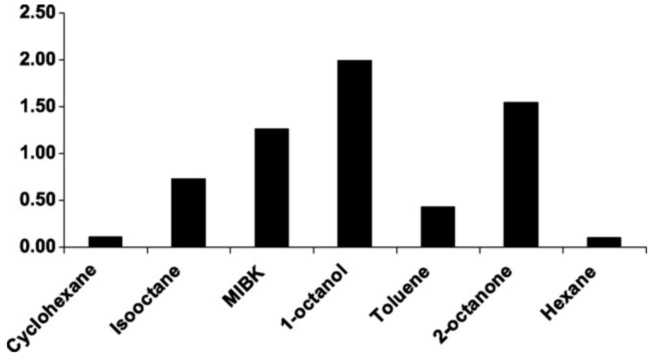

Fig. 3. Distribution coefficients of propionic acid between water and solvents used in this study.

one amine per *complex*, the loading increases with the increasing amine concentration. Systems that exhibit aggregation, i.e., formation of complexes with large numbers of acid and amine molecules, exhibit an abrupt increase in loading.

In this work, loading factors of all solvents decrease with increasing amine concentration, indicating that complexes include more than one amine per complex [22].

The values of the overall extraction constants, K_{11}, K_{12}, K_{23}, are calculated using Eq. (5) and presented in Table 2. In the case of proton-donating diluent (1-octanol) the extraction process can be described by the reactions:

$$(\mathrm{HA})^R + (\mathrm{R_2N})^E \rightleftarrows ((\mathrm{HA})\cdot(\mathrm{R_2N}))^E\ K_{11} \tag{9}$$

$$(\mathrm{HA})^R + 2(\mathrm{R_2N})^E \rightleftarrows ((\mathrm{HA})\cdot(\mathrm{R_2N})_2)^E\ K_{12} \tag{10}$$

The resulting acid: amine complexes are supposed to be stabilized due to hydrogen bonding with the diluent. Only the values of K_{11} and K_{12} for 1-octanol has been presented in Table 2.

In the case of non-proton donating diluents (hexane, cyclohexane, isooctane, toluene, 2-octanone and MIBK) the process can be described by the reactions:

$$2(\mathrm{HA})^R + 3(\mathrm{R_2N})^E \rightleftarrows ((\mathrm{HA})_2\cdot(\mathrm{R_2N})_3)^E\ K_{23} \tag{11}$$

Only the values of K_{11} and K_{12} for 1-octanol have been presented in Table 2. Reaction (11) can be understood as a result of three consecutive reactions—(9), (10) and (12)

$$(\mathrm{HA}\cdot(\mathrm{R_2N})_3)^E + ((\mathrm{HA})\cdot(\mathrm{R_2N})_2)^E \rightleftarrows ((\mathrm{HA})_2\cdot(\mathrm{R_2N})_5)^E\ K_A \tag{12}$$

The aggregation of highly polar primary acid: amine complexes according to reaction (12) is supposed to proceed almost completely. As the $((\mathrm{HA})\cdot(\mathrm{R_2N})_2)^E$ *complex* is the minor component in the studied range of concentrations, its presence is not indicated.

The distribution data of propionic acid between water and solvents used in this study (1-octanol, 2-octanone, cyclohexane, isooctane, hexane, cyclohexane and methyl isobutyl ketone (MIBK)) are presented in Table 3 and shown in Fig. 3.

5. Conclusions

This work examined the effect of diluents on the distribution of propionic acid between water and Amberlite LA-2. The diluents used were hexane, cyclohexane, isooctane, toluene, methyl isobutyl ketone (MIBK), 1-octanol and 2-octanone. The extraction of propionic acid with seven various solutions of amines increases the amine concentration with increasing amine. Among the diluents used in this study the largest distribution coefficients were obtained with MIBK.

The maximum extraction efficiencies for solvents used at maximum Amberlite LA-2 concentration are determined as

MIBK > 2-octanone > 1-octanol > toluene > isooctane > hexane > cyclohexane.

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