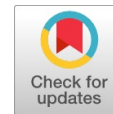# Recovery of Propionic Acid from Aqueous Phase using Alamine 336 in 1 - Octanol by Reactive Extraction

B. Sarath Babu, Priyanka Seerla Venkata

Abstract: The by-products of carboxylic acids are useful in chemical industry, pharmaceutical industry, preservations, drug delivery towards a more economical energy efficient technology. Physical and chemical equilibria results are presented as equilibrium distribution, distribution co -efficient, extraction efficiency, loading ratio and volume percentage by varying concentrations. The distribution of propionic acid between aqueous phase and organic phase reaches equilibrium over a concentration range of 0.009 kmol/m³ – 0.055 kmol/m³. The optimum TOA concentration is found to be 40% at which K_D value for 1 - octanol is 16.7. The modified separation increases with decrease in TOA & acid concentration and alamine loading with acid Z decreases with increase in alamine concentration in 1 – octanol. Optimum extraction efficiency is 43.82% for a conc. of 40% TOA at 0.2 kmol/m³

I. INTRODUCTION

Carboxylic acids which include propionic acid, succinic acid, lactic acid can be recovered or extracted by traditional methods which involves processes like acidification, crystallization and acidification by ion-exchange resins. However, the rapid industrialization of recent decades has resulted in a reduction or depletion of natural resources, and the growing concern for the environment has prompted a search for clean and environmentally friendly technologies [1]. The recovery of carboxylic acids, which provide a complex functional group of a solute from aqueous solution, using the reactive extraction method has become popular in recent years [2].
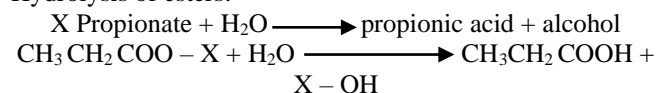
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Propionic acid is obtained mainly by three chemical routes [3][17]: Oxidation of propanol:

$$\text{Propanol} + O_2 \longrightarrow \text{Propionic acid}$$
$$CH_3 CH_2 CH_2OH + O_2 \longrightarrow CH_3 CH_2 COOH$$

Oxidation of propanol
$$\text{Propanol} + [\,O\,]\ (H_2SO_4/\ KMnO_4) \longrightarrow CH_3CH_2COOH$$

Hydrolysis of esters:
$$X\ \text{Propionate} + H_2O \longrightarrow \text{propionic acid} + \text{alcohol}$$
$$CH_3 CH_2 COO - X + H_2O \longrightarrow CH_3CH_2 COOH + X - OH$$
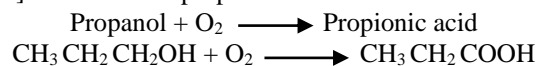
Propionic acid can also be commercially produced by oxidation of liquid phase propane or propionaldehyde. Propionic acid production by fermentative and chemical processes has not been economically competitive. Therefore it has not been used commercially [4]. Reactive extraction uses extractants from three different groups. There are several of them, such as solvation with carbon- and phosphorus-bonded oxygen-bearing extractants, proton transfer or ion-pairing creation with large molecular weight aliphatic amines and their salts, as well as others [5]. Tertiary amines have good extractabilities for carboxylic acids due to their higher molecular weight, high viscous and corrosive property [6]. Active & inactive diluents are two different categories of diluents. The extraction is improved by a variety of polar, active, proton- or electron-donating solvents [7]. Tertiary aliphatic amines are one of the several extractants for reactive extraction that are frequently utilised in the reactive extraction of carboxylic acids. Chain length aliphatic amines are rather poorly soluble in water without losing any extractant [8][13][14][15][16]. They are also thermostable, making distillation a viable method for regeneration. In general, tertiary amines are preferred to primary or secondary amines. With carboxylic acid, secondary amines may possibly form amides, losing both the product and the extractant [9 – 10].

Selecting the appropriate extractant is crucial for a reactive extraction procedure. he selectivity for the product in comparison to water and a high capacity for the product are its two most crucial properties. The separation factor and distribution coefficient serve as representations of these two needs. In order to overcome the solute's low activity in the aqueous phase, the binding between the extractant and the solute should also be robust, but not so strong as to make regeneration difficult or impossible [11]. This paper includes reactive extraction of propionic acid using alamine – 336 in 1 – octanol.
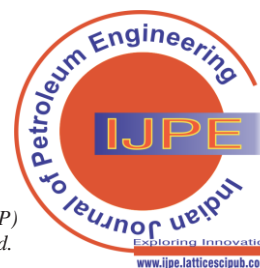
II. EXPERIMENTAL PROCEDURE

A. Materials

Alamine 336 (TOA) (98%), Propionic acid (99%), diluent 1-octanol (ACS Reagent (99%)). The solutions of various propionic acid concentrations were made using distilled water. Titration is done using NaOH and phenolphthalein indicator.

B. Apparatus

100 ml standard flask ,25 ml standard flask ,125 ml separating funnel, pH meter, rotating shakers, beakers, pipette, measuring jar.

C. Procedure

Initially 25 ml aqueous solution of required acid concentration was prepared in a conical flask. To this 25 ml of 1-octanol (organic phase) without tertiary amine was added for physical equilibrium. For chemical equilibrium the required quantity of tertiary amine ie., 10% ($0.2066 kmol/m^3$), 20% ($0.4132 kmol/m^3$), 30% ($0.6198 kmol/m^3$), 40% was taken in the 1-octanol. Experiments on physical and chemical equilibria were performed at 30°C, or normal temperature. Propionic acid's starting concentration was adjusted from 0.05 to 0.4 kmol/m3. Tri-octyl-amine concentrations of 10% ($0.2066 kmol/m^3$), 20% ($0.4132 kmol/m^3$), 30% ($0.6198 kmol/m^3$), and 40% in 1-octanol were studied at concentrations of 0.05, 0.1, 0.2, 0.3, and 0.4 $kmol/m^3$ of propionic acid.

Equal amounts (25 ml) of both aqueous and organic solutions are used in extraction experiments, which are run for 12 hours at a constant room temperature. At least two hours were given for the mixture to settle at the same temperature. pH of the aqueous phase was determined using a pH metre. Propionic acid aqueous solutions range in pH from 2.65 to 3.13. Based on the variations in densities, the organic and aqueous phases were separated after settling. A sample of 2 ml is used to estimate the quantity of acid in the aqueous phase using potentiometric titration with 0.05 N NaOH solutions & phenolphthalein as an indicator. The organic phase's acid concentration was estimated using mass balance. Different pH values from 3 to 7 were used in the chemical equilibrium studies [12].

III. RESULTS & DISCUSSION

A. Physical Equilibria

Equilibrium data are produced utilising 1-octanol as a diluent for the propionic acid physical extraction from the aqueous phase. Figure 1, 2 show the distribution of propionic acid between aqueous phase and the organic phase, at equilibrium, over the concentration range of 0.05 $kmol/m^3$ - 0.4 $kmol/m^3$. 1 – Octanol is polar in nature. $[PA]_{aq}$ increases with increase in $[PA]_{org}$.
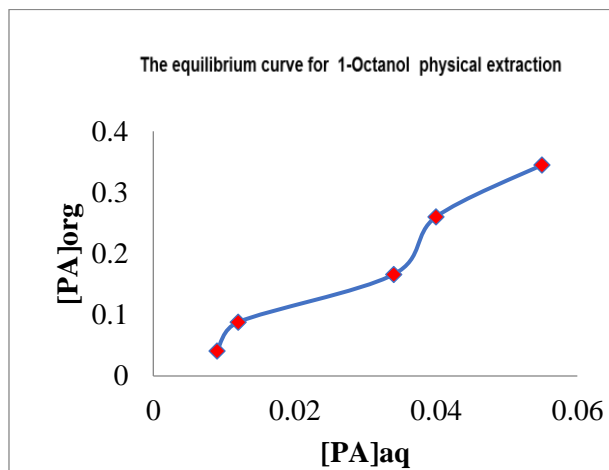

Fig. 1: Physical Equilibrium of (Water – Propionic Acid –1-Octanol)
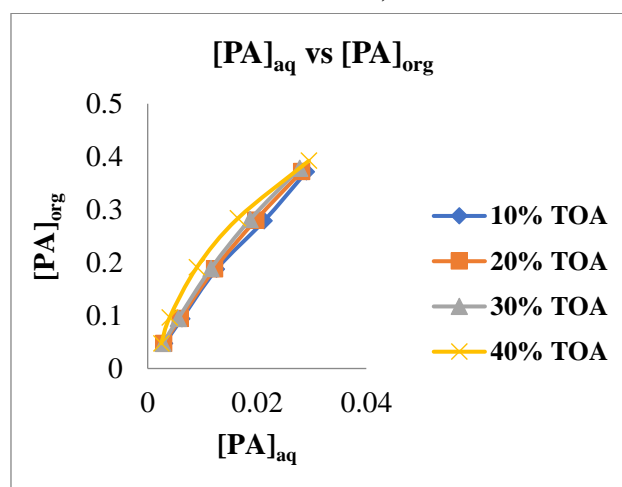

Fig. 2: Equilibrium for Reactive Extraction

Figure 1 shows in organic phase the variation of acid with aqueous phase for different concentrations of propionic acid from 0.05 $kmol/m^3$ to 0.4 $kmol/m^3$ in 1-octanol. It is observed that the increase in initial concentration of propionic acid increases the acid concentration in both aqueous phase and organic phase. This indicates that the extraction was clearly impacted by the tri-n-octyl amine in 1-octanol. At any acid concentration, with increasing tri-n-octyl amine concentration, more acid was extracted from aqueous solutions.

The physical extraction figure 1 show that the acid in organic phase for 0.05. 0.1, 0.2, 0.3, and 0.4 $kmol/m^3$ initial concentration of propionic acid are 0.04, 0.088, 0.166, 0.26 and 0.345$kmol/m^3$ respectively.

The chemical extraction figure 2 show that the acid in organic phase for the same initial concentrations of propionic acid and 10% TOA are 0.0469, 0.0937, 0.1874, 0.2789, and 0.372 $kmol/m^3$ respectively.

B. Chemical Equilibria
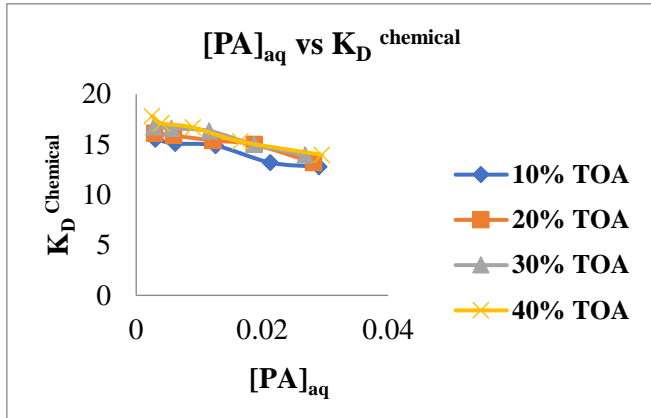
a. Distribution Coefficient

Fig. 3: Effect of Aqueous Concentration $[PA]_{aq}$ on $K_D^{Chemical}$

Alcohols are active diluents by inter molecular hydrogen bonding and also is 1-octanol is insoluble in water due to which the amount of acid extracted at any initial acid concentration from aqueous solution increases with increasing amine concentration and decreasing acid concentration. This can be clearly seen in figure 3.

b. Extraction Efficiency

Propionic acid concentration in aqueous medium has an impact on extraction effectiveness, as shown in Figure 4. When the propionic acid concentration is reduced, the level of extraction dramatically increases. The influence of aqueous concentration on extraction efficiency has been studied using various TOA concentrations, and it has been shown that the extraction efficiency improved as tri-n-octyl amine concentrations rose. This is brought on by the fact that when TOA concentration rises, the acid concentration in the aqueous phase decreases.
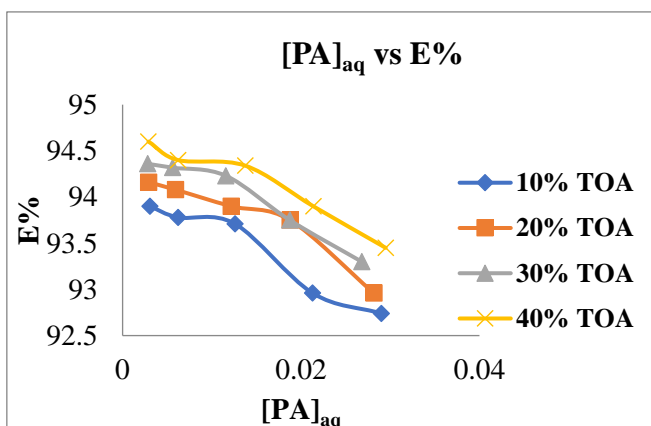

Fig. 4: Effect of Aqueous Concentration on Extraction Efficiency

c. Loading Ratio

The stoichiometry of the entire extraction method is determined by the loading ratio, Z, in the organic phase.
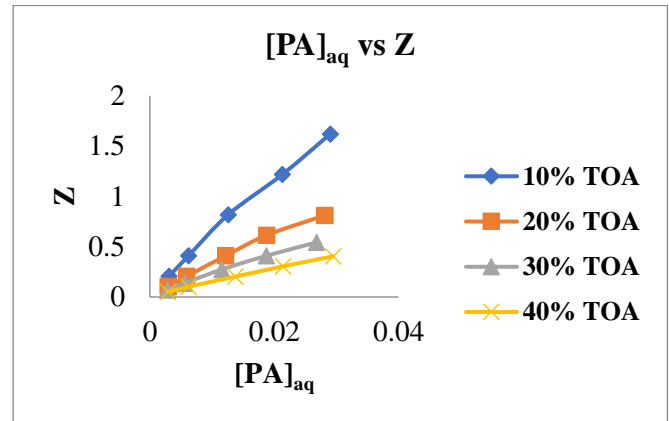

Fig. 5: Effect of Aqueous Concentration on Loading Ratio

It can be seen in Figure 5 that the loading ratio (Z) declined as tri-n-octyl amine concentrations increased. Only when complexes comprising more acid molecules form at lower tri-n-octyl amine concentrations does this occur. It is obvious that complexes containing a single amine develop since loading decreases as tri-n-octyl amine concentration increases.

d. Effect of Volume Percentage of TOA

The distribution coefficient first increased then decreased, It has increased for chemical extraction ($K_D^{Chemical}$) from 10 – 40% and decreased from 40 - 60% TOA in 1-octanol. The 40 volume % of TOA is considered to be the optimum because high K_D is obtained. Propionic acid is one of the by products from fermentation and effluent industries. The acid amine complex involve with a few acid molecules at higher concentrations of tri-n-octyl amine.
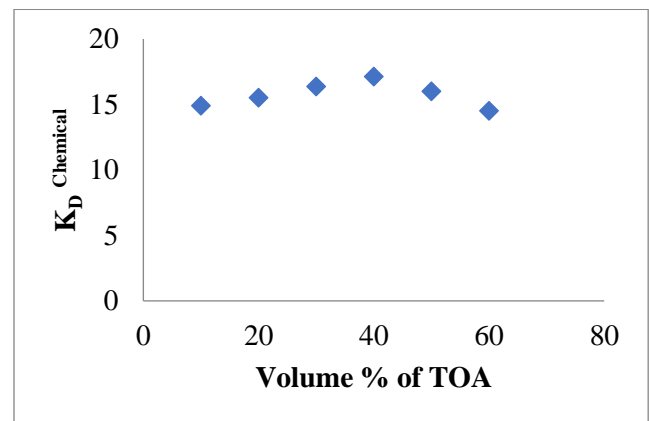
Fig. 6: Effect of Volume % for Extraction of 0.2 kmol/m3 Propionic Acid

e. Effect of pH

pH was calculated using pH meter. Propionic acid extraction employing 40% TOA in 1-octanol is shown in figure 7. Since the pH of the fermentation broth changes as acid is created, it is crucial to research how pH affects the extraction of propionic acid at varied pH values. It is discovered that extraction is relatively low at pH 5 of acid when pH is changed from 3.0 to 7.

Fig. 7: Effect of pH

The cause is greater pH levels. Acid is mostly present in its dissociated state, while TOA only removes acid in its undissociated form. As a result, K_D ^{Chemical} is quite low. For a 30% TOA in 1-octanol, it can be shown that a rise in pH causes a progressive reduction in K_D ^{Chemical} with almo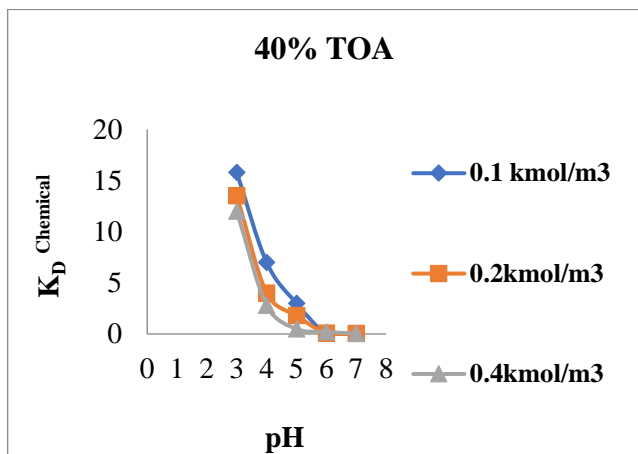st no extraction at pH 6. For 40% TOA in 1-octanol, however, $K_D^{Chemical}$ decreases sharply when pH is raised above natural pH; beyond that, the reduction is more gradual and slower. The most likely explanation for this is that when pH rises, the proportion of undissociated acid decreases. The excessive amount of TOA present caused the existing bonding sites to become cluttered, which resulted in a very low acid partition from the aqueous phase.

f.   Modified Separation Factor

From figure 9 the S_f observed is 0.5172 at initial concentration of acid 0.1 kmol/m^3 and TOA concentration of 30% in 1-octanol. From figure 10 for same concentration of acid and 40% TOA in 1-octanol, the S_f is found 0.5157 which is lower that 30% TOA concentration.
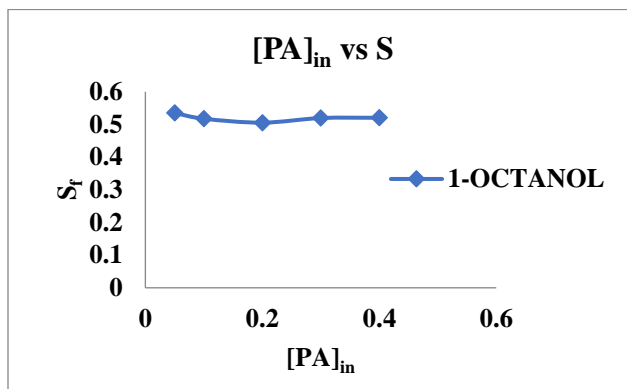

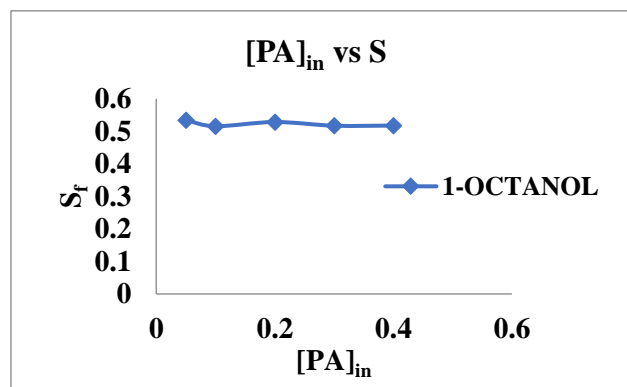**Fig. 8: Effect of Acid Concentration ([PA]in) on Sf using 30% Alamine**

Fig. 9: Effect of Acid Conc. ([PA]in) on Sf using 40% Alamine

Figures 8 & 9 depict how the modified separation factor affects the initial acid concentration. With increasing acid content, a reduction in the relative proportion between physical interaction and chemical reaction, indicated by S, was seen. While loading was observed to decrease with higher TOA concentrations in 1-octanol.

Table 1: Physical Equilibrium of (Water – Propionic Acid –1-Octanol)

$[PA]_{aq}$	$[PA]_{org}$
0.009	0.041
0.012	0.088
0.034	0.166
0.04	0.26
0.055	0.345

Table 2: Equilibrium for Reactive Extraction of Propionic Acid

10% (T)		20% (T)		30% (T)		40% (T)	
$[PA]_{aq}$	$[PA]_{org}$	$[PA]_{aq}$	$[PA]_{org}$	$[PA]_{aq}$	$[PA]_{org}$	$[PA]_{aq}$	$[PA]_{org}$
0.00305	0.04695	0.00292	0.04708	0.0028	0.0472	0.0025	0.0475
0.00621	0.09378	0.00592	0.09408	0.0056	0.0944	0.004	0.096
0.01259	0.1874	0.01219	0.1878	0.01158	0.18884	0.009	0.191
0.0213	0.2789	0.01876	0.28124	0.01874	0.28126	0.0165	0.2835
0.029	0.3710	0.02816	0.37184	0.0268	0.3732	0.02949	0.3705

Table 3: Effect of Aqueous Concentration on KD With Varying TOA Concentration in 1-Octanol

10% (T)		20% (T)		30% (T)		40% (T)	
$[PA]_{aq}$	$K_D^{chemical}$	$[PA]_{aq}$	$K_D^{chemical}$	$[PA]_{aq}$	$K_D^{chemical}$	$[PA]_{aq}$	$K_D^{chemical}$
0.00305	15.5	0.00292	16.12	0.0028	16.75	0.0025	17.8
0.00621	15.09	0.00592	15.89	0.0056	16.6	0.004	17.1
0.01259	14.89	0.01219	15.4	0.01158	16.35	0.009	16.7
0.0213	13.2	0.01876	14.99	0.01874	15.01	0.0165	15.3
0.029	12.78	0.02816	13.2	0.0268	13.94	0.02949	13.97

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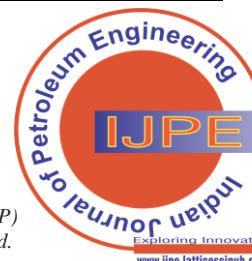
Table 4: Effect of Aqueous Concentration on Extraction Efficiency

10% (T)		20% (T)		30% (T)		40% (T)	
$[PA]_{aq}$	E%	$[PA]_{aq}$	E%	$[PA]_{aq}$	E%	$[PA]_{aq}$	E%
0.00305	93.90	0.00292	94.16	0.0028	94.36	0.0025	94.6
0.00621	93.78	0.00592	94.08	0.0056	94.318	0.004	94.4
0.01259	93.71	0.01219	93.9	0.01158	94.23	0.009	94.34
0.0213	92.96	0.01876	93.75	0.01874	93.75	0.0165	93.9
0.029	92.74	0.02816	92.96	0.0268	93.3	0.02949	93.45

Table 5: Effect of Aqueous Concentration on Loading Ratio

10% (T)		20% (T)		30% (T)		40% (T)	
$[PA]_{aq}$ Kmol/m^3	Z	$[PA]_{aq}$ Kmol/m^3	Z	$[PA]_{aq}$ Kmol/m^3	Z	$[PA]_{aq}$ Kmol/m^3	Z
0.00305	0.2050	0.00292	0.1028	0.0028	0.0687	0.0025	0.051
0.00621	0.4095	0.00592	0.2054	0.0056	0.1374	0.004	0.1023
0.01259	0.8184	0.01219	0.4099	0.01158	0.2743	0.009	0.2033
0.0213	1.2179	0.01876	0.6139	0.01874	0.4094	0.0165	0.3041
0.029	1.6200	0.02816	0.8120	0.0268	0.543	0.02949	0.4045

Table 6: Effect of pH on KDChemical for different acid Conc. of Propionic Acid using 40% TOA

$[PA]_{in}$	pH	$[PA]_{aq}$	$[PA]_{org}$	$K_D^{chemical}$
0.1	3	0.00489	0.0951	19.45
	4	0.00943	0.09057	8.8
	5	0.0227	0.07727	2.9
	6	0.053	0.0474	0.09
	7	0.095	0.00476	0.05

$[PA]_{in}$	pH	$[PA]_{aq}$	$[PA]_{org}$	$K_D^{chemical}$
0.2	3	0.0133	0.1867	14
	4	0.02597	0.17402	6.6
	5	0.04762	0.15238	3.2
	6	0.18349	0.01651	0.09
	7	0.19048	0.009524	0.05

$[PA]_{in}$	pH	$[PA]_{aq}$	$[PA]_{org}$	$K_D^{chemical}$
0.4	3	0.0308	0.3692	12
	4	0.05479	0.3452	6.3
	5	0.08333	0.3167	3.8
	6	0.36697	0.03303	0.0-9
	7	0.3809	0.01905	0.05

IV. CONCLUSION

Propionic acid is obtained by reactive extraction using alamine 336 in 1 – Octanol. Physical and chemical equilibria for propionic acid by Tri – n octyl amine in 1 - Octanol as a diluent have been determined. . The optimum TOA concentration is found to be 40% at which K_D value for 1 - octanol is 16.7. The modified separation increases with decrease in TOA & acid concentration and loading of TOA with acid Z decreases with increase in TOA concentration in 1 – octanol. Optimum extraction efficiency is 43.82% for a conc. of 40% TOA at 0.2 kmol/m^3. In future different carboxylic acids can be extracted using alamine 336 or aliquat in different diluents due to the high applications of carboxylic acids in various different fields.

DECLARATION STATEMENT

Funding	No, I did not receive.
Conflicts of Interest	No conflicts of interest to the best of our knowledge.
Ethical Approval and Consent to Participate	No, the article does not require ethical approval and consent to participate with evidence.
Availability of Data and Material	Not relevant
Authors Contributions	All authors have equal participation in this article.

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AUTHORS PROFILE

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