

Reactive Extraction of Propionic Acid: Equilibrium and Kinetic Studies

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Abstract: Reactive extraction is an efficient, economical and environmentally friendly method for separating acids from waste streams. The separation of organic residues from aqueous waste streams released from industries is essential from the point of view of pollution control and recovery of useful materials. Propionic acid has become an important feedstock in chemical industries for manufacturing biodegradable polymers and synthetic resins. The disposal of wastewater containing propionic acid has been recognized as a significant expense to the industry and the environment. This study used reactive extraction to separate propionic acid from an aqueous solution using Tri-n-Pentyl Amine and Tri-n-Butyl Amine with different diluents. Kinetic variables such as order of reaction, mass transfer coefficient and rate constants were determined by conducting kinetic studies using a stirred cell in a batch-type reactive system. Physical equilibrium experiments showed that 1-Octanol has the highest extractable strength compared to chloroform and dimethylene chloride. The distribution coefficient of chemical extraction is higher than that of physical extraction. Effect of acid (0.1 to 0.4 N), amine concentration (10% to 40%) is noticed in terms of Distribution coefficient K_D in the range of 3 to 19 for TPA and K_D in the range of 1.5 to 5.67 for TBA at 0.1N. The Equilibrium complexation constant (K_{E1}) was calculated using the loading ratio (Z) at 10% to 40% amine in 1-octanol. At 40% maximum Equilibrium complexation constants for TPA K_{E1} =10.28 and TBA K_{E1} =5.94 were obtained. The reaction regime depended on the Hatta number value, with TPA being instantaneous and TBA being a fast reaction. The enhancement factors for TPA and TBA were found to be 5.1 and 3.8, respectively.

Keywords: Equilibrium, Kinetics, Reactive Extraction, Propionic Acid

1. Introduction

Propionic acid is a clear liquid with a strong odour. Food preservatives, pharmaceutical formulations, textiles, and rubber industries are using this organic molecule [1]. Due to the depletion of petroleum resources and environmental contamination, propionic acid is generated through the microbial fermentation method. Propionic acid is separated using a method known as reactive extraction [2]. Reactive extraction is a method for separating propionic acid from an aqueous solution using a suitable solvent and diluent system that is fast, cost-effective, and process-intensified [3].

Diffusion and solubilization processes form an acid amine complex at the interface between the extractant and the solvent [4].

Aliphatic amines have excellent extractability, yield, and

propionic acid recovery due to their hydrophobic nature. Active diluents such as 1-octanol, chloroform, and dimethylene chloride aid in maintaining the complex's stability and preventing re-extraction [5, 20].

The significance of this paper is to determine the effect of chemical reactions on the specific rate of mass transfer using equilibrium and kinetic studies. It's made by diluting propionic acid (solute), tri-n-pentyl amine, and tri-n-butyl amine (extractants) with 1-octanol, chloroform, and dimethylene chloride (Active diluents).

2. Materials and Methods

2.1. Materials

Propionic acid [$C_3H_6O_2$] (Merck, 99%) and purified water are used to make an aqueous solution with a concentration

range of 0.1 to 0.4 kmol/m³. To make the organic solution, extractants and diluents were used. As the extractants like tri-n-butyl amine [(CH₃)₃CNH₂] (Finar, 98%) and tri-n-pentyl amine [CH₃(CH₂)₄N] (Aldrich, 99%) were used.

Diluents included 1-octanol [CH₃(CH₂)₇OH] (Sisco, 98%), chloroform [CHCl₃], and dimethylene chloride [CH₂Cl₂] (Molychem, 98%). The NaOH (85%) was used as reagent grade. P^H=8–10 phenolphthalein indicator (Hi media, 85%). All of the chemicals were used without being purified initially.

2.2. Methods

2.2.1. Equilibrium Studies

The tests were carried out at room temperature (306 K) and atmospheric pressure (101.32 KPa). A separate conical flask (100ml) containing equivalent volumes of aqueous and organic phases were held in a temperature-controlled shaker bath (supplied by REMI laboratory instruments). The two phases were left to stabilise until they reached equilibrium (1hour). An acid-base titration with NaOH and phenolphthalein as an indicator was used to assess the acid concentration. Physical equilibrium data can also be obtained in the organic phase by using diluent. The law of mass action determined the acid concentration in the organic phase.

2.2.2. Kinetic Studies

A 500ml cylindrical vessel (Diameter=80mm, length=115mm) with a double paddle impeller (Diameter=8mm, thickness=2mm, the space between impellers=1mm) is used to perform the kinetic studies. The kinetic studies were carried out in a 500ml cylindrical vessel (diameter 80mm, length 115mm) with a double paddle impeller (diameter 8mm, thickness 2mm, the gap between impellers 1mm). In a stirred cell, 100ml of the first aqueous phase is taken. The four-blade impeller is positioned 1mm above and 1mm below the interface without disrupting it, an equivalent volume (100ml) of the organic phase was placed in a cell. Low RPMs were taken in between the range of 100 to 120, so that interface could not get disrupted.

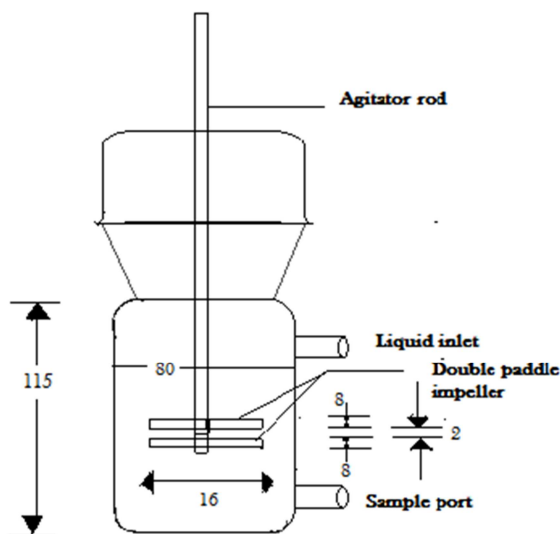


Figure 1. Schematic diagram of a stirred cell for kinetic studies (All dimensions are in mm).

To determine the concentration of propionic acid in the aqueous phase, an aqueous sample (10ml) has been taken each 10 minutes with a syringe from the sample port and titrated with 0.5N NaOH. The sample was collected from the aqueous solution until it came to a steady state which time was taken 1 hour. Mass balance was used to investigate Propionic acid in the organic phase. The process is repeated with different amines (Tri-n-pentylamine and Tri-n-butylamine) and diluents (1-Octanol, Chloroform, and dimethylene chloride) at different Propionic acid concentrations.

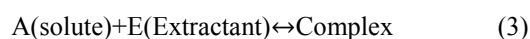
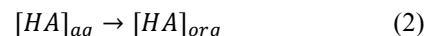
Whereas, the reaction is reversible, the possibility with backward extraction of acid molecules from organic to an aqueous phase. Kinetic studies allow for the testing of back mixing of propionic acid by calculating the initial specific reaction rate, R_{HA} (mole/m²s), which is driven only by the forward reaction.

$$R_{HA} = \left(\frac{V_{org}}{A_C} \frac{dC_{org}}{dt} \right)_{at t=0} \quad (1)$$

2.3. Theory of Reactive Extraction

As a promising method for the recovery of propionic acid, reactive extraction with a predefined extraction, giving more distribution coefficient has been expected [6]. In reactive extraction, the concentration of the aqueous and organic phase, which becomes complex, the extractant, and the temperature are all involved.

The water and complex form when the hydroxyl group of the acid reacts with the hydrogen atoms in the extraction. Propionic acid moves from the an aqueous phase to the interface, where it interacts with the extractant to form a complex, which is then dissolved in the diluent [7, 18].



3. Results and Discussions

3.1. Extraction Equilibria

3.1.1. Physical Equilibrium Isotherms

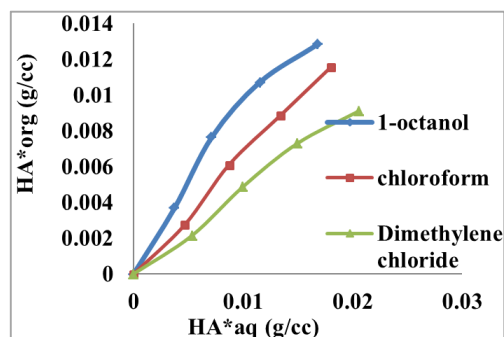


Figure 2. Variety of diluents for extraction of Propionic acid with Physical equilibrium isotherms.

Physical equilibrium isotherms in terms of (g/cc) were determined by performing physical equilibrium, diverse acid

concentrations with a variety of diluents. It refers to the quantity of acid in g diffused in 1 L of solution. From Figure 2. It is found that 1-octanol has more capability to extract the acid in aqueous phase followed by chloroform and dimethylene chloride.

3.1.2. Chemical Equilibrium Isotherms

Between the extractant and the solute, an acid-amine

complex forms in a chemical equilibrium reaction. Isotherms were plotted for various extractant concentrations at different acid concentrations. Figure 3 shows that as the amine concentration rises, the rate of diffusivity of propionic acid increases from aqueous to organic solution for both TPA and TBA. It should also be noticed that TPA has a higher extractive capacity than TBA.

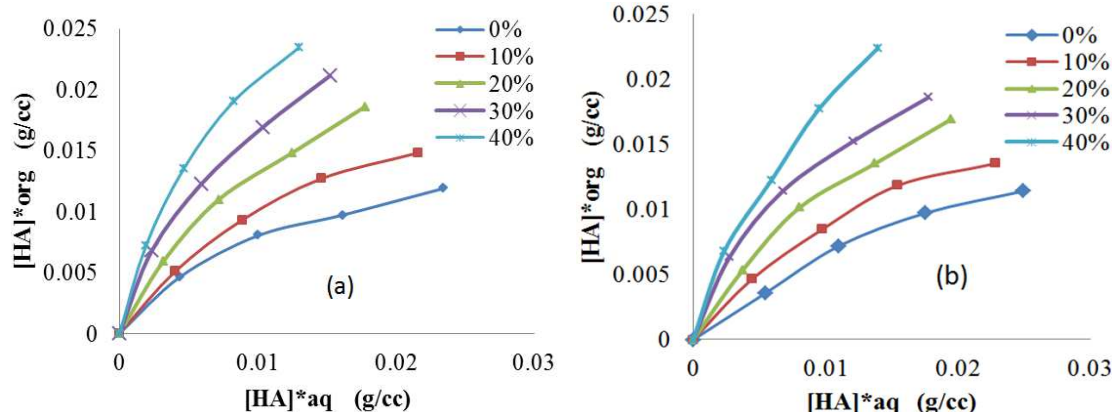


Figure 3. Chemical equilibrium isotherms of Propionic acid at 0.1 Kmole/m³ for reactive extraction at different concentrations of (a) TPA = 0%, 10%, 20%, 30%, 40% (=0, 0.343, 0.687, 1.03, 1.4 Kmole/m³) and (b) TBA = 0%, 10%, 20%, 30%, 40% (=0, 0.4219, 0.843, 1.3, 1.7 Kmole/m³) in 1-octanol.

3.1.3. Loading Ratio

The Z (loading ratio) determines the amount of propionic acid concentration that can be loaded in the organic phase (Tri-n-pentyl amine in 1-octanol).

$$Z = \frac{[HA]_{org}}{[E]_0} \quad (4)$$

Z is determined by the propionic acid's extractability (acid-amine interaction strength) and its aqueous concentration, but it is unaffected by the amine concentration. A plot of $Z/(1-Z)$ versus $[HA]_{org}$ gives a straight line with a slope as a constant equilibrium complexation for very low

loading ratios of propionic acid in the organic phase ($Z < 0.5$), i.e. for (1:1) acid-amine complex. [9].

Table 1. Equilibrium complexation is constant for the reactive extraction of (1:1) propionic acid in 1-octanol with different diluents at 40 percent TPA and TBA.

S.No.	Diluent	K_{E1} for TPA in 1-octanol	K_{E1} for TBA in 1-octanol
1	1-octanol	10.28	5.94
2	Chloroform	5.86	4.04
3	Dimethylene chloride	3.61	2.77

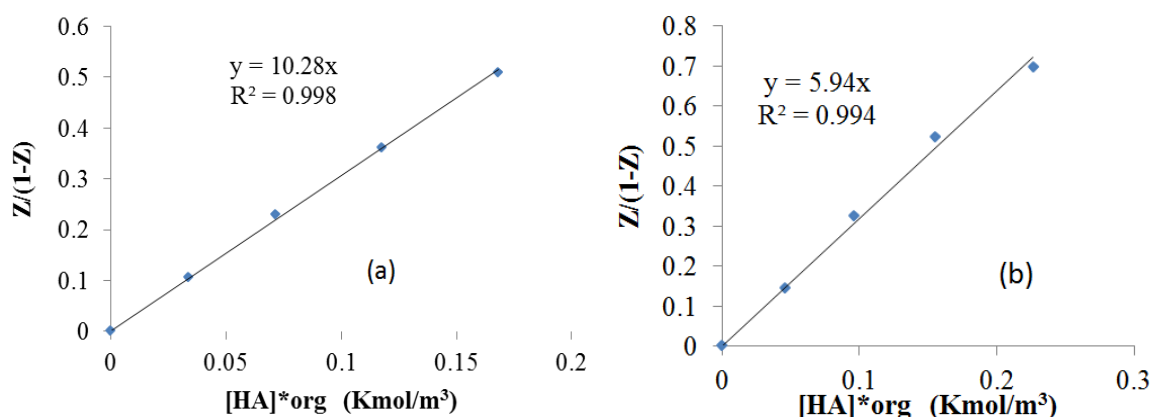


Figure 4. A plot of $\frac{Z}{(1-Z)}$ Vs. $[HA]_{org}$ for the estimation of equilibrium complexation constant (1:1) Propionic acid at (a) 40% Tri-n-pentyl amine and (b) 40% Tri-n-butyl amine in 1-octanol.

Figure 4 shows that only (1:1) acid-amine complex formation is possible due to the high equilibrium propionic acid concentration in the organic phase. A non-collinear

pattern is obtained from Figure 5, indicating that propionic acid molecules in the organic phase are insufficient for the formation of the (2:1) propionic acid-amine complex.

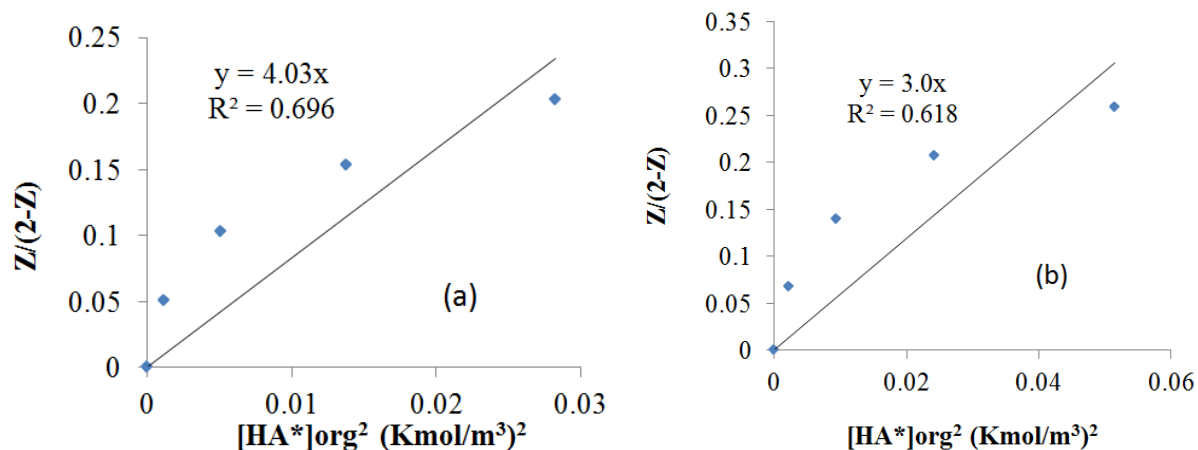


Figure 5. A plot of $\frac{Z}{(2-Z)}$ Vs. $[HA]^*_{org}$ for the estimation of equilibrium complexation in constant (2:1) Propionic acid at (a) 40% Tri-n-pentyl amine and (b) 40% Tri-n-butyl amine in 1-octanol.

3.2. Distribution Coefficient

The amount of propionic acid content that has diffused from the aqueous phase to the organic phase can be determined using the distribution coefficient (K_D). It is a measure of the solute distribution for the two phases [8, 17].

$$K_D = \frac{[HA]_{org}}{[HA]_{aq}} \quad (5)$$

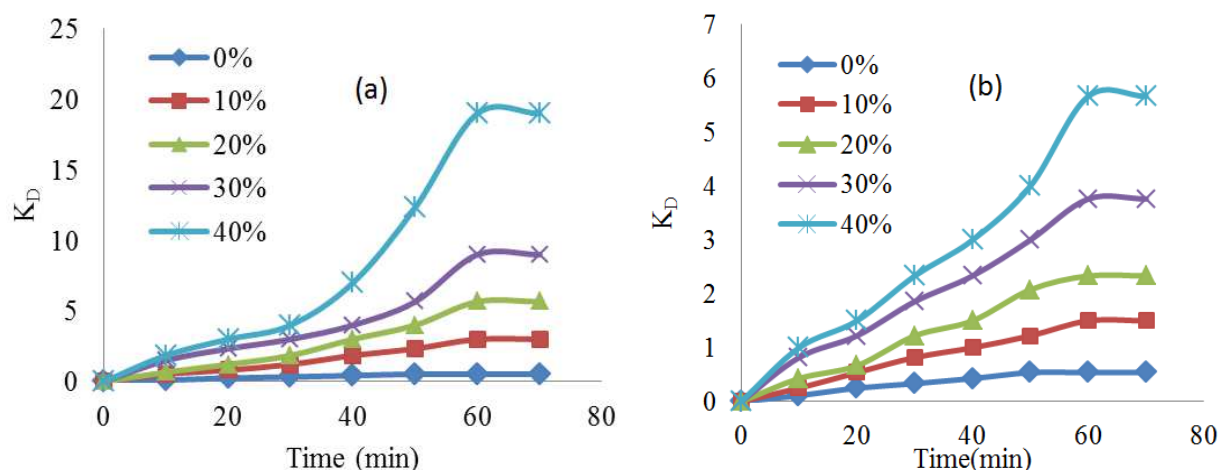


Figure 6. Effect of distribution coefficient in time for different concentrations (a) Tri-n-pentyl amine and (b) Tri-n-butyl amine (0%, 10%, 20%, 30% and 40% $Kmol/m^3$) with 1-octanol at 0.1 $Kmol/m^3$ Propionic acid concentration.

As shown in figure 6, with increasing the time, distribution coefficient is increased to enhance the TPA and TBA concentrations, when it reaches the stabilised state it remains constant. Since there are more carbon atoms available in TPA with 1-octanol $K_D = 19$ than, TBA along with 1-octanol $K_D = 5.67$, there is more potential for forming a complex with propionic acid.

3.3. Effect of Initial Acid Concentration

The variation of the distribution coefficient was calculated with various concentrations of TBA and TPA diluted in

different diluents at different concentrations (0.1 to 0.4 $Kmol/m^3$) of Propionic acid.

From figure 7, we can deduce that. As the propionic acid concentration increases, the distribution coefficient (K_D) decreases. At a low propionic acid concentration range (0.1 $Kmol/m^3$), The majority ratio of propionic acid molecules is bound to amine $[HA]_{org}$, remaining propionic acid molecules stay in aqueous phase $[HA]_{aq}$. It is forecasted to be higher than the ratio of (0.4 $Kmol/m^3$) at the higher concentration level in the aqueous phase, implying that K_D decreases drastically with an increase in acid concentration.

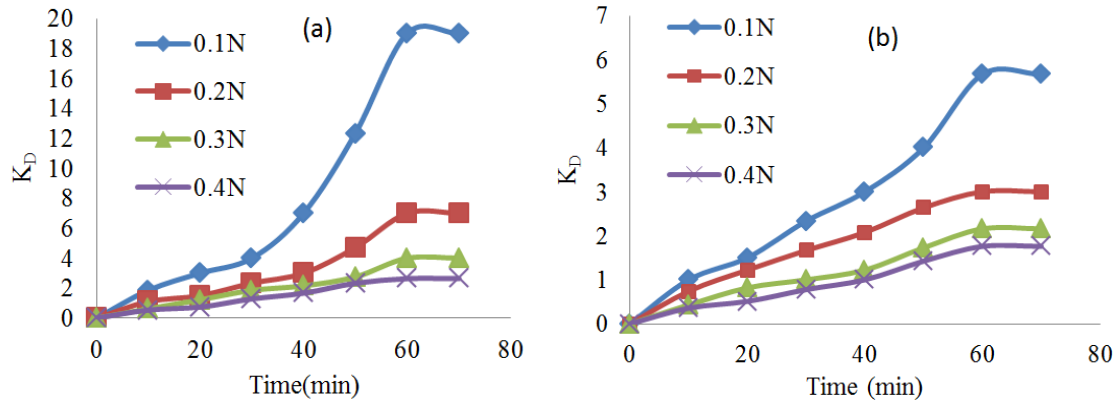


Figure 7. Variation of distribution coefficient with time for different concentrations of Propionic acid and concentration of (a) 40% (1.4 Kmole/m³) TPA and (b) 40% (1.7 Kmole/m³) TBA in 1-octanol.

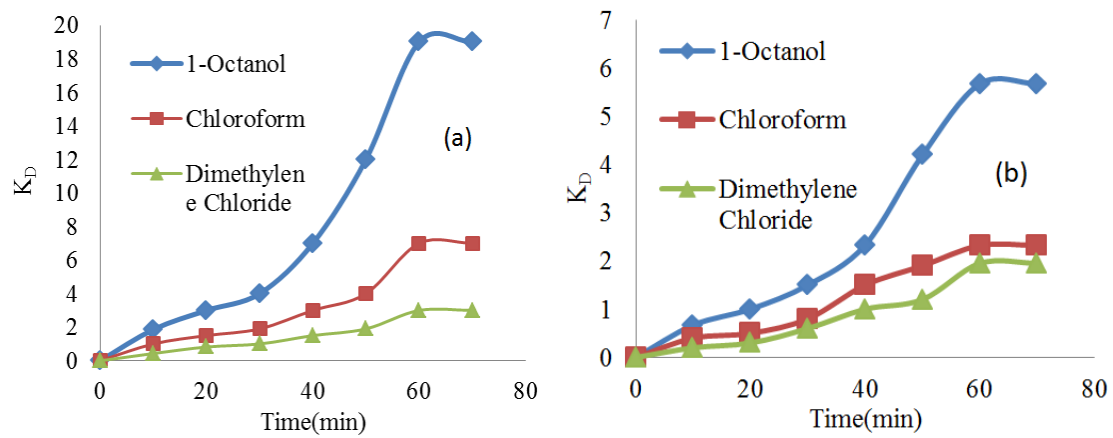


Figure 8. Effect of diluents on distribution coefficient for 0.1 Kmole/m³ Propionic acid (a) 40% (1.4 Kmole/m³) of TPA and (b) 40% (1.7 Kmole/m³) TBA.

Because of its high polarity and acid extractability, 1-octanol has the highest distribution coefficient as compared to chloroform and dimethylene chloride, as shown in Figure 8.

3.4. Effect of Extraction Efficiency

The ratio of acid concentration to initial acid concentration can be used to determine the extraction efficiency of propionic acid [10].

$$\eta = \frac{K_D}{1+K_D} \quad (6)$$

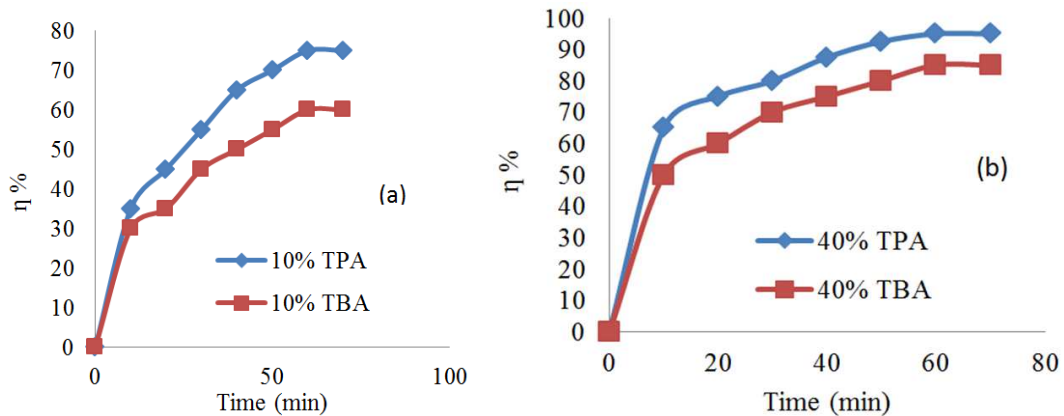


Figure 9. Effect on extractants of reactive extraction for Tri-n-pentyl amine and Tri-n-butyl amine concentrations of (a) 10% and (b) 40% for 0.1 Kmole/m³ propionic acid concentration.

From figure 9 the extraction efficiency of propionic acid improves as the amine concentration increases for both TPA and TBA with different diluents. At higher amine concentrations, TPA with 1-octanol (94%) and TBA with 1-octanol (85%) achieve maximum propionic acid extraction efficiency (0.1 Km³/m³).

3.5. Kinetic Studies

3.5.1. Physical Mass Transfer Coefficient

The mass transfer coefficient K_L (m²/s) is determined to validate the reactive system's regime.

It can be done by conducting propionic acid physical extraction in a batch style reactive method using only pure diluent [10, 14]. For a batch style reactive device, the differential mass balance can be written as

$$V_{org} \frac{dC_{org}}{dt} = K_L A_c (C^*_{org} - C_{org}) \quad (7)$$

By integrating the above equation, we obtained as follows

$$\ln \left(\frac{C^*_{org}}{C^*_{org} - C_{org}} \right) = \frac{K_L A_c t}{V_{org}} \quad (8)$$

A plot between $\ln \left(\frac{C^*_{org}}{C^*_{org} - C_{org}} \right)$ Versus $\frac{A_c t}{V_{org}}$ Yields a straight line and the slope gives the value of $K_L = 3.5 \times 10^{-6} \text{ m}^2/\text{s}$.

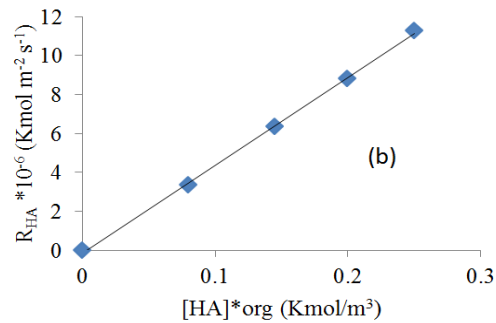
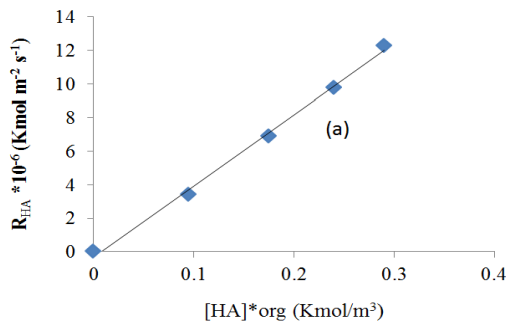


Figure 11. The result of the initial Propionic acid concentration on the basic rate of extraction in 1-octanol for the reactive extraction of 0.1Kmol/m³ Propionic acid with (a) TPA=40% =1.4 Km³/m³ and (b) TBA=40% =1.7 Km³/m³.

3.5.3. Reaction Order Concerning the Initial Concentration of Amine

Order can be found by calculating the specific rate of extraction for various concentrations of TPA and TBA at the

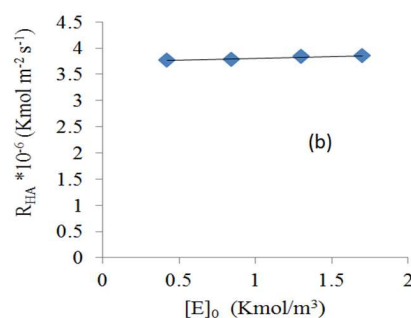
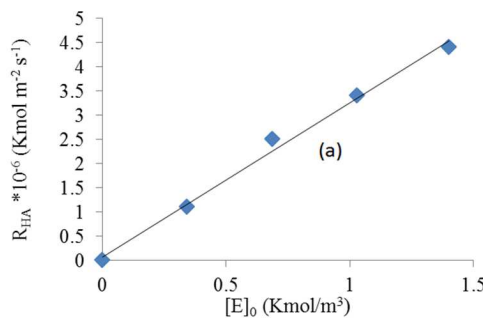


Figure 12. Effect of the initial amine concentration of 0.1 Km³/m³ of Propionic acid on the particular rate of extraction for the reactive extraction of Propionic acid with (a) Tri-n-pentyl amine and (b) Tri-n-butyl amine in 1-octanol.

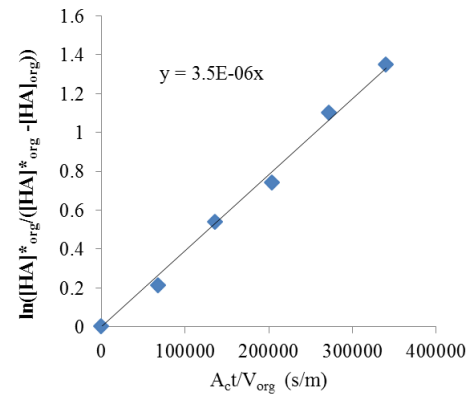


Figure 10. The overall mass transfer coefficient is estimated At 0.1 Km³/m³ propionic acid in 1-octanol.

3.5.2. Reaction Order Concerning the Initial Concentration of Acid

Regression analysis can be used to determine the order of the reaction by measuring the specific rate of reaction at different propionic acid concentrations (0.1 to 0.4 Km³/m³) at a given amine concentration (40 percent). The line in Figure 11 passes through the origin with a slope of m=1 for both TPA and TBA. The order of TPA and TBA in 1-Octanol is found to be the first order in terms of initial acid (propionic acid) concentration.

necessary propionic acid concentration, i.e. at 0.1 Km³/m³, which can be used to find order. It is clear from Figure 12 that TPA has an amine effect in 1-octanol (n=1), but TBA has does not effect 1-octanol (n=0).

3.5.4. Rate Constant

For a given m , n order of an acid amine complex, Doraiswamy and Sharma had given the equation to calculate the rate constant. K_{mn} ($\text{mol}/\text{m}^2\text{s}$) [12].

$$R_{HA} = [HA]_{org}^* (\sqrt{D_A K_{mn} [HA]^{m-1} [E]_0^n}) \quad (9)$$

Where D_A is the Propionic acid diffusion coefficient in 1-octanol. It can be calculated using Doraiswamy and Sharma's equation [11, 16].

$$D_A = 7.4 \times 10^{-12} \left(\frac{T \sqrt{\varphi M_B}}{\mu^{0.6}_{acid}} \right) \quad (10)$$

Where Association factor (φ), Molecular Weight (M_B) and Viscosity (μ) for 1-octanol are $\varphi = 1$, $M_B = 130.23$ kg/kmol, $\mu = 7.36 \times 10^{-3}$ kg/m-s, Molar Volume of Propionic

acid is $0.0814 \text{ m}^3/\text{Kmol}$, D_A can be obtained as $1.56 \times 10^{-9} \text{ m}^2/\text{s}$.

The rate of expression can be reduced to for the obtained order (m, n).

$$R_A = [HA]_{org}^* (\sqrt{D_A K_2 [E]_0^n}) \text{ For } m=1, n=1 \quad (11)$$

$$R_A = [HA]_{org}^* (\sqrt{D_A K_1}) \text{ For } m=1, n=0 \quad (12)$$

From Figure 13 the value of the second-order rate constant was estimated to be $0.1547 \text{ m}^3 \text{ kmol}^{-1} \text{ s}^{-1}$ by performing a regression analysis between R_{HA}^2 and $D_A [E]_0 [HA]^*_{org}{}^2$, (K_2) gives a straight line with a slope. Plotting a graph of R_{HA}^2 versus $[HA]^*{}^2$ (K_1) gives a straight line with a slope of 0.093 s^{-1} , which is the value of the first-order rate constant. As compared to Tri-n-butyl amine with 1-octanol, the rate constant for Tri-n-pentyl amine is found to be higher.

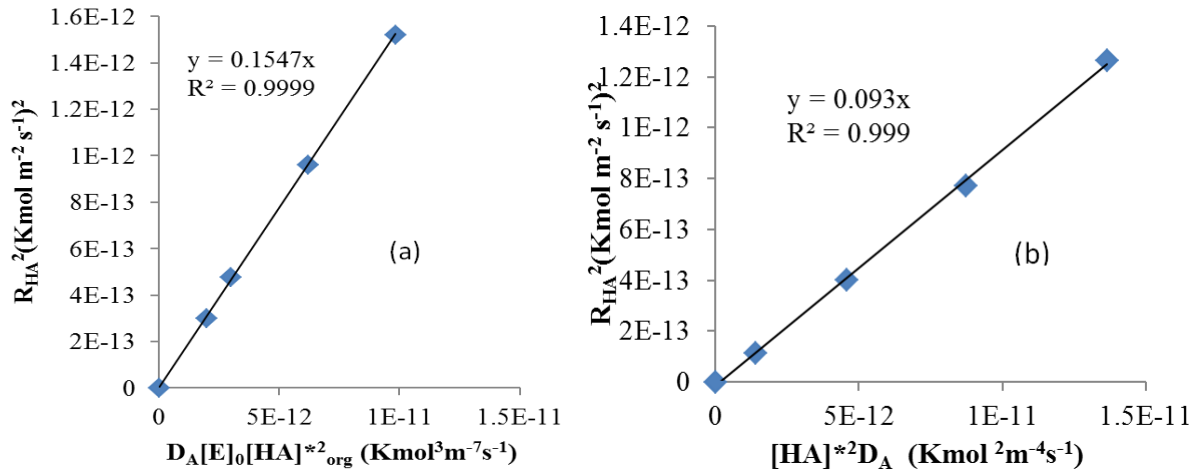


Figure 13. Determination of second-order rate constant for (a) 40% TPA = $1.4 \text{ kmol}/\text{m}^3$ and first-order rate constant for (b) 40% TBA = $1.7 \text{ kmol}/\text{m}^3$ in 1-octanol at different Propionic acid concentrations ranging from 0.1N to 0.4N.

3.5.5. Hatta Number (M_H)

The four types of regimes have been classified by Doraiswamy and Sharma. (Very slow, slow, fast and instantaneous). The value of the Hatta Number can be used to determine the form of a regime for a given reaction order (m, n). [12, 15]

$$M_H = \left(\frac{\frac{2}{m+1} K_{mn} D_A [HA]^*{}^{m-1} [E]_0^n}{K_L} \right) \quad (13)$$

Hatta Number varies from 5.5 to 13.6 for various concentrations of TPA (0.343 to 1.4) Kmol/m^3 with 1-octanol, and from 3.1 to 8.4 for TBA (0.4219 to 1.7) Kmol/m^3 . The reaction is in the instantaneous or fast reaction regime since $M_H \gg 1$. To confirm the type of regime for a given reaction order the necessary condition for the validity of the regime is

$$M_H \gg \frac{[E]_0}{Z[HA]^*} \left(\sqrt{\frac{D_B}{D_A}} \right) D_A K_2 [E]_0 \gg K_L^2 \text{ for } m=1, n=1$$

$$\text{Instantaneous (regime 4)} \quad (14)$$

$$M_H \ll \frac{[E]_0}{Z[HA]^*} D_A K_1 \gg K_L^2 \text{ for } m=1, n=0 \text{ Fast reaction (regime 3)} \quad (15)$$

The value of $\frac{[E]_0}{Z[HA]^*} \left(\sqrt{\frac{D_B}{D_A}} \right)$ varies from 2 to 4.5 and $D_A K_2 [E]_0 \gg K_L^2$ for TPA and $\frac{[E]_0}{Z[HA]^*}$ lies in between 0.9 to 2.6 and $D_A K_1 \gg K_L^2$ for TBA. Since the criteria conditions for the desired regimes were met. As a result, for TPA, an instantaneous reaction occurs at the interface in the organic phase of the reactive system, whereas for TBA, a fast reaction occurs simultaneously in the film.

3.5.6. Enhancement Factor

The enhancement factor, which is described as follows, can be used to measure the effect of a reaction on the pure mass transfer of propionic acid from the aqueous to the organic phase [13, 19].

$$\Phi = \frac{R_{HA}}{K_L [HA]^*_{org}} \quad (16)$$

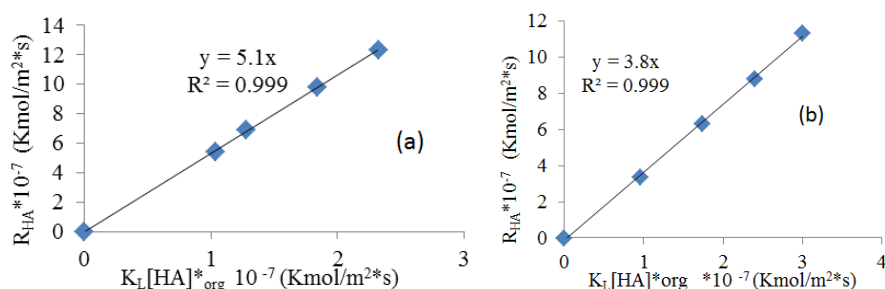


Figure 14. Estimation of enhancement factor in 1-octanol using 0.1 Kmol/m^3 propionic acid concentration with (a) 40% Tri-n-pentyl amine = 1.4 kmol/m^3 and (b) 40% Tri-n-butyl amine = 1.7 kmol/m^3 .

The Figure 14 is used to investigated that TPA has an enormous enhancement factor of 5.1, while TBA enhancement factor rate is 3.8. As a result, the effect of the propionic acid extraction reaction with TPA in 1-octanol is higher.

4. Conclusions

The Reactive extraction process has been used to explore the equilibrium and kinetics of propionic acid. The distribution coefficient of chemical extraction is found to be higher than that of physical extraction. As compared to chloroform and dimethylene chloride, 1-octanol, as an active

diluent, is recommended as the best diluent. In 1-octanol, it is also determined that both TPA and TBA have an acid effect, but TBA has no amine effect.

The value of K_L was found to be $3.56 \cdot 10^{-6} \text{ m/s}$ using the differential mass balance equation. $K_2 = 0.1547 \text{ m}^3 \text{ kmol}^{-1} \text{ s}^{-1}$ for TPA and $K_1 = 0.09 \text{ s}^{-1}$ for TBA were found to be the rate constants. The reaction regime was found to be dependent on the value of Hatta number, with TPA being instantaneous and TBA is being fast reaction. Since the enhancement factors for TPA and TBA were found to be 5.1 and 3.8, respectively. As a result, the effect of the reaction for propionic acid is greater than for TPA with 1-octanol.

List of Abbreviations

The following are the list of abbreviations used in the paper.

$[HA]_{org}$	Concentration of Propionic acid in organic phase (Kmol/m^3)
$[HA]_{aq}$	Concentration of Propionic acid in aqueous phase (Kmol/m^3)
$[HA]_{in}$	Initial Propionic acid concentration (Kmol/m^3)
$[HA]_{org}^*$	Concentration of Propionic acid in organic phase at equilibrium (Kmol/m^3)
$[E_0]$	Initial Amine concentration (Kmol/m^3)
K_D	Distribution coefficient
η	Extraction efficiency
Z	Loading ratio
D_A	Diffusivity of Propionic acid in 1-octanol (m^2/s)
R_{HA}	Specific rate of extraction of Propionic acid ($\text{Kmol/m}^2\text{s}$)
K_L	Mass transfer coefficient (m/s)
M_H	Hatta modulus
Φ	Enhancement factor
$[A_c]$	Cross sectional area of the stirred cell (m^2)
V_{org}	Volume phase (m^3)
T	Time of extraction (min)
K_2	Second order rate constant ($\text{m}^3/\text{Kmol*s}$)
K_1	First order rate constant (s^{-1})
K_{E1}	Equilibrium complexation constant for 1:1 Acid amine complex
K_{E2}	Equilibrium complexation constant for 2:1 Acid amine complex

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