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RESEARCH ARTICLE

# Liquid – Liquid Extraction of Hydrochloric Acid from Aqueous Solutions by Tri-n-dodecylamine and Tri-n-octylamine / diluents

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***Abstract***

*The extraction of HCl from aqueous solutions using two types of long-chain tertiary amines tri-n- dodecylamine/ kerosene and tri-n-octylamine/ kerosene in the presence of octanol-1 or tributyl phosphate as modifier has been studied. The effect of mixing time, HCl concentration in the aqueous phase, amine concentration, organic to aqueous phase ratio, modifier percentage in the organic phase, NaCl concentration in the aqueous phase and temperature were studied. The effect of some diluents such as: toluene, benzene and xylene on HCl extraction was also investigated and found to follow the order:*

*Benzene > toluene > kerosene + 10% octanol-1 ≈ xylene*

*Tri-n-dodecylamine and tri-n-octylamine showed a great affinity towards HCl in all tested diluents. Both amines (TDA and TOA) can be successfully used for HCl recovery from aqueous acidic solutions.*

*Water was found to be effective to strip HCl from the acid loaded organic phase and more than 95% of HCl can be recovered in three stripping stages at* $V_{aq}/V_{org} = 20/1$ *and 75 °C.*

***Keywords***

*extraction, hydrochloric acid, tri-n-dodecylamine/ kerosene, tri-n-octylamine, octanol-1, tri-n-butylphosphate*

## 1 Introduction

Hydrochloric acid is an important chemical and widely used in a variety of industries and chemical processes and in some cases as a very successful substitute in processes that otherwise use sulphuric acid. The largest applications of HCl are: Steel pickling, oil well acidizing, food manufacturing, plastics, pharmaceuticals, organic chemical synthesis, and ore processing [1-4].

A large amount of acidic waste water which is listed as a hazardous waste will be generated from hydrometallurgical and acid pickling industrial units. So, the recovery of HCl from its aqueous streams is crucial and needed to prevent environmental pollution and to recycle the recovered acid.

Tertiary amines such as: Tri-n- dodecyl amine, tri-n-octylamine, Alamine 308 (tri-isooctylamine), TEHA (tri-2-ethyl hexyl amine), Alamine 336 (a mixture of trioctyl/decyl amine) have been proven to be effective extractant to recover HCl and $H_2SO_4$ from aqueous solutions [5-8]. The extraction reaction of HCl by tertiary amines can be written as follows:

$$R_3N_{(org)} + HCl_{(aq)} \rightleftarrows R_3NHCl_{(org)} \qquad (1)$$

In general, tertiary amines, due to their physical properties they must always be used in the form of solutions in organic diluents to reduce their viscosity and small amount of high long-chain alcohol (isodecanol, octanol-1, etc.) must be added to prevent third phase formation [9, 10]. As an alternative to long chain alcohol, a third phase inhibitor is to add a high aromatic content diluent to the amines (+ 90% aromatic), but the aromatic diluents produce environmental problems and known as carcinogenic [11].

It has been found that diluents, especially those with functional groups can affect the extraction behavior of amine significantly. The stoichiometry of solute: amine complex, loading of amine as well as the third phase formation are influenced by the diluents [12].

Many studies which have been reported in the literature are devoted to study the extraction of HCl from aqueous phases. Nelson et al. [13] investigated the equilibrium between tri-n-octylamine and hydrochloric acid and the equilibrium constant of the extraction reaction TOA + HCl ⇆ TOAHCl was found to be $5.6.10^6$.

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Another study of two phase equilibria between triisooctylamine dissolved in several organic diluents and dilute aqueous solution of HCl was reported by A. S. Vieux and N. Rulagengwa [14], they found that, in 1, 2-dichloroethan, dichloromethane, 1,2- dichlorobenzene and chloroform the ammonium salts usually exist as ion pairs. In the low dielectric constant diluents, benzene, xylene, and carbon tetrachloride, the ion pairs aggregate. The semi-thermodynamic formation constant for the extraction of the ion paired tri-isooctylammonium chloride depends on the polarity or dielectric constant of the diluent and on the chemical nature of the diluent.

S. Brandani et al. [15] studied the extraction of strong mineral acids (HCl and $H_2SO_4$) from aqueous solutions using secondary amine Amberlite LA-2. Experimental measurements were performed with aqueous solutions in the concentration range 0.01−0.5 M. the organic phase comprised 0.5 M Amberlite in toluene, they found that $H_2SO_4$ has greater affinity than HCl vs Amberlite LA-2.

Alamine 336 was successfully used by Agarwal et al [5] and found to be very effective to recover $H_2SO_4$ from aqueous solutions generated during the process of pickling of steel at (30−60 °C). Stripping of the loaded acid was done with distilled water at 60 °C.

R. Banda et al [6] studied the extraction of HCl from chloride leach solution of hydro-disulfurization catalyst using trioctylamine, Alamine 330, Alamine 308 and TEHA. He found that stripping of HCl from the loaded extractant TEHA is easier when compared with other tested tertiary amines and a pure HCl was recovered.

In the present paper we report the partition of hydrochloric acid between water and tri-n- dodecylamine and tri-n- octylamine containing octanol-1 in kerosene and all parameters influencing the extraction process such as: stirring time, acid concentration in the aqueous phase, extractant concentration, modifier (octanol-1or TBP) percentage in the organic phase, organic to aqueous phase ratio, and also the effect of some diluents (benzene, toluene and xylene) on the extraction has been investigated.

Finally the stripping of hydrochloric acid from the organic phase by water has been also studied and the optimum operating conditions for HCl extraction and stripping were determined.

## 2 Experimental

### 2.1 Reagent

All the chemicals used in this study were of analytical grade reagent. Tri-n-dodecylamine (TDA) and tri-n- octylamine (TOA) were obtained from MERCK and FUKA with a purity of 95% and 98% respectively and used as extractants without any further treatment. The used diluent was the kerosene PEMCO SOLV 110 from PEMCO Chemicals company. The density of the kerosene was 800-810 kg/m$^3$ at 15 °C, with a boiling point range 200-250 °C, and flash point >70 °C, with aromatic content of max 0.5% (v/v). Octanol-1 and tri-n- butyl phosphate were used as modifiers was obtained from MERCK with a purity of 99% for each.

Sodium hydroxide ampoules (0.1N and 1N) from BDH, were used for the preparation of 0.1 N and 1 N of NaOH by diluting the content of the ampoules to 1 L with double distilled water.

The aqueous solutions of hydrochloric acid were prepared by diluting concentrated hydrochloric acid (37%) from MERCK, with double distilled water.

The organic phases of TDA and TOA were prepared by diluting them with kerosene, benzene, toluene and xylene. Octanol-1 was added to the organic phases when kerosene used as diluents to prevent third phase formation.

### 2.2 Extraction procedure

Solvent extraction experiments were carried out at a constant temperature by stirring equal volume (25 mL) each of hydrochloric acid solution and organic phase in a Stirring Bath Thermostat with a temperature constancy = ± 0.1 °C; After equilibration, the phases were allowed to settle and separated.

The initial and the equilibrium concentrations of hydrochloric acid in the aqueous phases and the equilibrium concentration of hydrochloric acid in the organic phases were determined by simple titration with 0.1N or 1N of NaOH using an automatic potentiometric titrator (AT-420N-WIN) from KYOTO ELECTRONICS, Japan. It is important to note that, the organic phases were titrated in an aqueous medium of (80% (v/v) absolute ethanol/water) in order to obtain a homogenous phase during the titration. The relative standard deviation of HCl determination in all experiments conducted in this work was in the range of 0.1% to 3%.

The distribution ratio ($K_d$), and the yield of hydrochloric acid extraction ($Y_{HCl}$ %) were calculated as follows:

$$K_d = \frac{Total\ \ concentration\ \ of\ \ HCl\ \ in\ \ the\ \ organic\ \ phase\ \ at\ \ the\ \ equilibrium\ \ (M)}{Total\ \ Concentration\ \ of\ \ HCl\ \ in\ \ the\ \ aqueous\ \ phase\ \ at\ \ the\ \ equilibrium\ \ (M)}$$

$$= \frac{[HCl]_{org}}{[HCl]_{aq}}$$

$$Y_{HCl}\% = \frac{[HCl]_{org}.V_{org}}{[HCl]_i.V_{aq}} \times 100$$

Where $V_{aq}$ and $V_{org}$ refers to the volume of aqueous and organic phase respectively and $[HCl]_i$ , $[HC]_{aq}$ and $[HCl]_{org}$ refers to the concentration of hydrochloric acid in the aqueous phase (initial and equilibrium) and organic phase respectively.

## 3 Results and Discussion

### 3.1 Effect of contact time

The kinetics of hydrochloric acid extraction was studied by equilibrating Equal volumes of 0.2 M HCl with 0.1 M TDA/kerosene containing 10% octanol-1 at 25 °C for various time intervals. The results obtained are shown in Fig.1. in the form of the distribution ratio of hydrochloric acid against time. It is clear that 180 seconds is the minimum time required to reach the equilibrium.

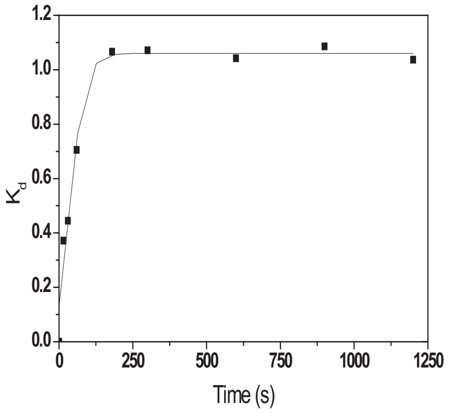

**Fig. 1** Effect of time on HCl extraction with TDA.
$[TDA]_i$ = 0.1 M/kerosene + 10 v/v% octanol-1, $[HCl]_i$ = 0.2 M.
$V_{org}/V_{aq}$ = 1 , t = 25 °C, time = 0−1200 s.

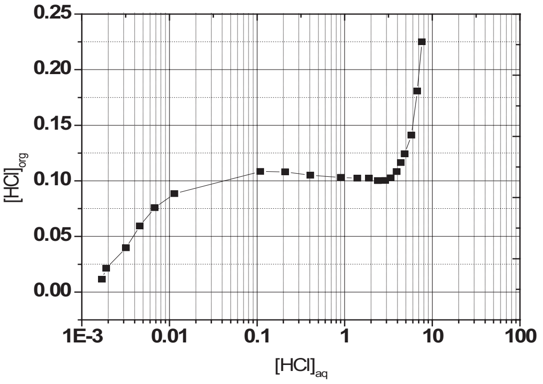

**Fig. 2** Variation of HCl concentration in the organic phase vs. HCl concentration in the aqueous phase.
$[TDA]_i$ = 0.1 M/kerosene + 10 v/v% octanol-1,
$[HCl]_i$ = 0.01–8 M. $V_{org}/V_{aq}$ = 1 , t = 25 °C.

### 3.2 Effect of HCl concentration in the aqueous phase

The extraction of hydrochloric acid in the range of (0.01−8 M) by 0.1 M tri-n-dodecylamine /kerosene containing 10% octanol-1 as modifier at an organic to aqueous phase ratio ($V_{org}/V_{aq}$ = 1), and at 25 °C was examined. The results are shown in Fig. 2. It is obvious that the concentration of hydrochloric acid in the organic phase $[HCl]_{org}$ was increasing in accordance with Le Chatelier's principle with the increase of hydrochloric acid in the aqueous phase $[HCl]_{aq}$, until $[HCl]_{org}$ reaches the value of 0.1 M, then no changes of $[HCl]_{org}$ was observed in the range of acidity ($[HCl]_{aq}$ = 0.2−3.5M) and the molar ratio of the extracted HCl concentration to the initial tri-n-dodecylamine concentration was equal to 1 ($[HC]_{org}/[TDA]_i \approx 1$). So it can be concluded that the nature of extracted species within this range of acidity will be in the form of $R_3N.HCl$. Afterwards, $[HCl]_{org}$ restarted to increase again with the increase of $[HCl]_{aq}$ from 3.5−8 M, which means that other species like $R_3N.(HCl)_2$ and $R_3N.(HCl)_3$ will be formed ($1<[HCl]_{org}/[TDA]_i<3$).

### 3.3 Effect of TDA and TOA /diluents on HCl extraction

An aqueous phase of 0.5 M of hydrochloric acid was given contact with different concentrations of TDA and TOA diluted in different diluents (kerosene, benzene, toluene and xylene) at the following operating conditions ($V_{org}/V_{aq}$ = 1, t = 40 °C). The results are presented in Fig. 3. It was observed that the distribution ratio $K_d$ is increasing with the increase of amine concentration in the organic phase. It is very important to note that the type of amine and diluents used had a clear effect on the extraction of hydrochloric acid. The yield of hydrochloric acid extraction by TOA is greater than TDA and the effect of diluents on hydrochloric acid extraction for both extractants can be summarized as follows:

Benzene > toluene > kerosene +10% octanol-1 ≈ xylene

Although the yield of hydrochloric acid extraction by TDA and TOA was improved by using benzene and toluene as diluents and there is no need to add a modifier to the organic phase, but it is not recommended to use aromatic diluents in an industrial scale because they are known to be carcinogenic and can produce environmental problems. Consequently using kerosene containing octanol-1 as modifier is advisable for its advantages, lower operation cost and lower toxicity.

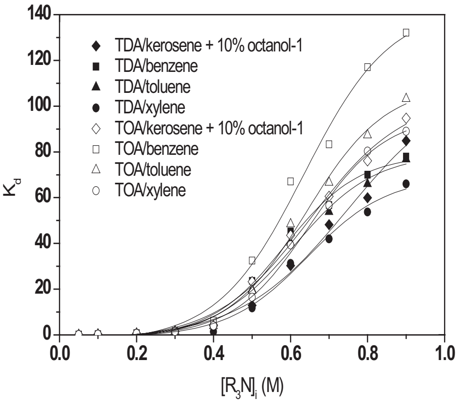

**Fig. 3** Variation of $K_d$ vs. initial amine concentration / different diluents.
◆ $[TDA]_i$ = (0.05−0.9M)+10% octanol-1/kerosene ,
■ $[TDA]_i$ = (0.05−0.9M)/benzene
▲ $[TDA]_i$ = (0.05−0.9M)/toluene, ● $[TDA]_i$ = (0.05−0.9M)/xylene.
◇ $[TOA]_i$ = (0.05−0.9M)+10% octanol-1/kerosene ,
□ $[TOA]_i$ = (0.05−0.9M)/benzene.
Δ $[TOA]_i$ = (0.05−0.9M)/toluene , ○ $[TOA]_i$ = (0.05−0.9M)/xylene.
$[HCl]_i$ = 0.5 M , $V_{org}/V_{aq}$ =1, t = 40 °C.

### 3.4 Effect of modifier (octanol-1, TBP) on HCl extraction

Two kind of modifiers octanol-1 and tributyl phosphate were tested in order to prevent third phase formation and to see their effect on HCl extraction by TDA/kerosene. The extraction experiments were carried out by mixing an aqueous solution of 0.5 M HCl with 0.1 M TDA/kerosene and 0.5 M TDA/kerosene containing different percentage of octanol-1 and TBP ranging from 5−35% (v/v), at the following operating conditions

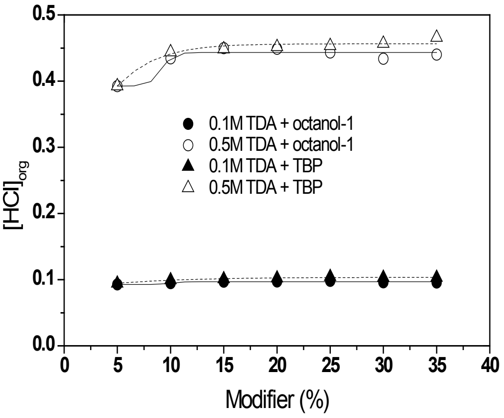

**Fig. 4** Variation of $[HCl]_{org}$ vs. octanol-1 and TBP percentage in the organic phase.
● $[TDA]_i$ = 0.1 M/kerosene + (5−35% v/v octanol−1).
○ $[TDA]_i$ = 0.5 M/kerosene + (5−35% v/v octanol−1).
▲ $[TDA]_i$ = 0.1 M/kerosene + (5−35% v/v TBP).
∆ $[TDA]_i$ = 0.5 M/kerosene + (5−35% v/v TBP).
$[HCl]_i$ = 0.5 M , $V_{org}/V_{aq}$ = 1, t = 40 °C.

($V_{org}$/ $V_{aq}$ = 1, t = 40 °C). The results were represented in Fig. 4. In the form of $[HCl]_{org}$ against modifier percentage in the organic phase. It can be seen from Fig. 4 that octanol-1 and TBP have a very small positive effect on HCl extraction and TBP has a little bit greater effect than octanol-1, especially at high amine concentration 0.5M TDA/kerosene. Thus, 10% (v/v) of octanol-1 or TBP is mandatory to be added to tri-n- dodecylamine to prevent third phase formation and accelerate phase separation.

### 3.5 Effect of phase ratio on HCl extraction

The effect of phase ratio on the extraction of HCl by TDA/ kerosene was done by mixing an aqueous solution of 0.5 M HCl with 0.1 M TDA/kerosene containing 10%(v/v) octanol-1 and 0.5 M TDA/kerosene + 10%(v/v) octanol-1 at different organic to aqueous phase ratios ($V_{org}/V_{aq}$) ranging from 0.5 to 3. The results presented in Fig. 5. In the form of HCl extraction yield ($Y_{HCl}$ %) against $V_{org}/V_{aq}$ ratio. It is obvious that the yield of HCl extraction increases with the increase of $V_{org}/V_{aq}$ ratio, and about 98% of HCl is extracted in one stage by 0.5 M TDA/kerosene +10% octanol-1 at $V_{org}/V_{aq}$ = 1.5. Consequently 0.5 M TDA/ kerosene +10%(v/v) octanol-1 was found to be very effective for the extraction of HCl and $V_{org}/V_{aq}$ = 1.5 is the best phase ratio to be used for economical and phase separation reasons.

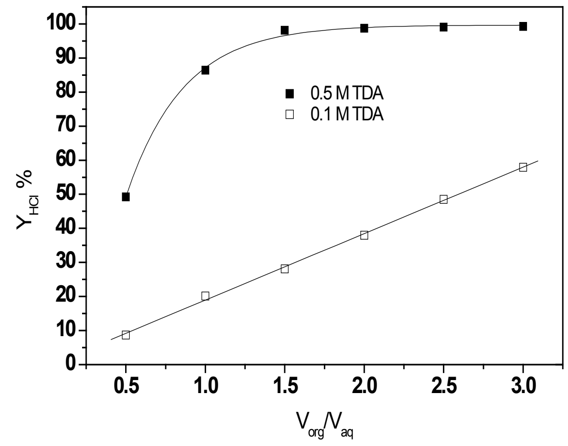

**Fig. 5** Variation of $Y_{HCl}$% vs. $V_{org}/V_{aq}$.
□ $[TDA]_i$ = 0.1 M /kerosene + 10 (v/v)% octanol-1.
■ $[TDA]_i$ = 0.5 M /kerosene + 10 (v/v)% octanol-1.
$[HCl]_i$ = 0.5 M , $V_{org}/V_{aq}$ = 0.5−3, t = 40 °C.

### 3.6 Effect of temperature on HCl extraction

The effect of temperature has been studied by mixing an aqueous feed solution of 0.5 M HCl with 0.1 M TDA/kerosene + 10%(v/v) octanol-1 at $V_{org}/V_{aq}$ = 1 at different temperatures (27−52 °C). The results presented in Fig. 6. In the form of Log $K_d$ vs 1/T and $Y_{HCl}$ (%) vs T, indicate that the yield of extraction of HCl decreases slowly with increasing temperature, that means the extraction reaction of HCl with tri-n-dodecylamine is exothermic. The value of enthalpy change of the extraction reaction ∆H° was calculated using van 't Hoff equation ($LogK_d = −∆H/2.303 RT + C$) from the slop of the straight line obtained by plotting $LogK_d$ against 1/T and found to be -4.1 kJ/mol.

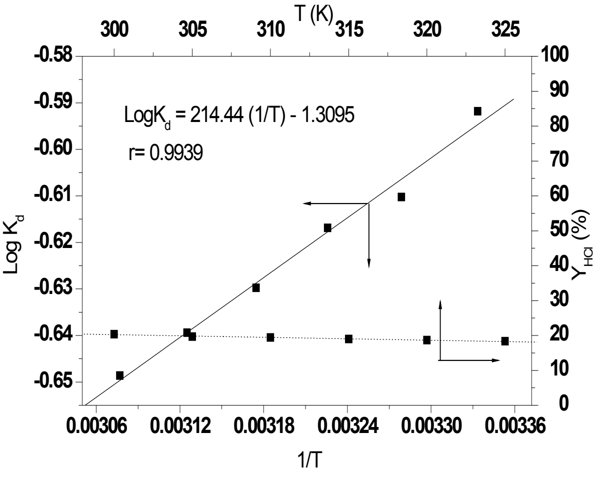

**Fig. 6** Effect of temperature on HCl extraction.
$[TDA]_i$ = 0.1 M/kerosene + 10% (v/v) octanol-1,
$[HCl]^i_{aq}$ = 0.5 M. $V_{org}/V_{aq}$ = 1 , T = (300−325 K).

### 3.7 Effect of NaCl concentration on HCl extraction

To study the effect of NaCl on hydrochloric extraction, a series of aqueous solutions containing 0.5 M HCl and different concentrations of NaCl ranging from 0.2 to 1 mol/L were given contact with 0.5 M TDA/kerosene + 10% (v/v) octanol-1 and 0.1 M TDA/kerosene + 10 % (v/v) octanol-1 at the following operation condition ($V_{org}/V_{aq}$ = 1, t = 40 °C). The results presented in Fig. 7. In the form of $Y_{HCl}$ % against NaCl concentration in the aqueous phase indicated that no effect of NaCl content on HCl extraction was observed.

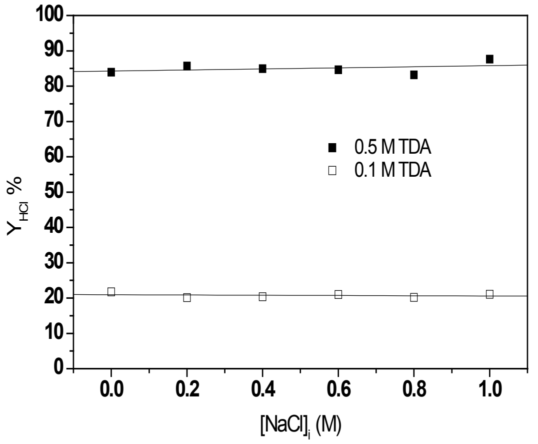

**Fig. 7** Effect of NaCl concentration in the aqueous phase on HCl extraction.
■ $[TDA]_i$ = 0.5 M/kerosene + 10 % (v/v) octanol-1,
□ $[TDA]_i$ = 0.1 M/kerosene + 10 % (v/v) octanol-1,
$[HCl]_i$ = 0.5 M, $[NaCl]_i$ = 0.2 – 1 mol/L, $V_{org}/V_{aq}$ = 1, t = 40 °C

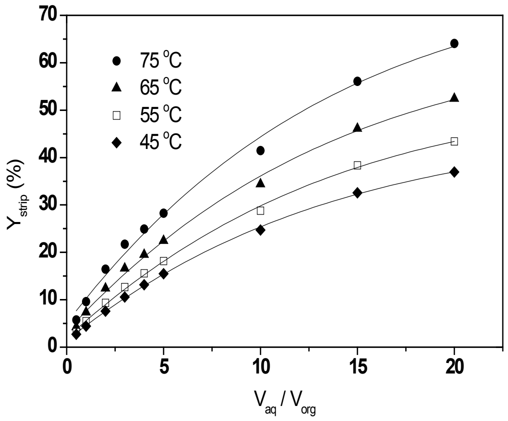

**Fig. 8** Effect of $V_{aq}/V_{org}$ ratio and temperature on HCl stripping from0.5 M TDA/kerosene + 10 % (v/v) octanol-1.
$[HCl]^i{}_{org}$ = 0.465 M, mixing time = 20 min,
$V_{aq}/V_{org}$ = 0.5–20. t = ♦ 45 °C, □ 55 °C, ▲ 65 °C, ● 75 °C.

### 3.8 Stripping of HCl from the loaded TDA/kerosene +10%(v/v) octanol-1

To investigate the ability of stripping hydrochloric acid from the loaded organic phase of 0.5 M TDA/kerosene + 10% (v/v) octanol-1 and regenerate the extractant, many stripping experiments were carried out by mixing the acid loaded organic phase with double distilled water at different organic to aqueous phase ratios ranging from 0.5 to 20 and at four different temperatures (45, 55, 65 and 75 °C). The results presented in Fig. 8. Show that the yield of HCl stripping is increasing with the increase of $V_{aq}/V_{org}$ ratio and temperature. The best operating conditions to recover about 64% of HCl from the loaded organic phase in just one contact were found to be ($V_{aq}$/ $V_{org}$ = 20, t = 75 °C) and in three successive contacts, more than 95% of HCl will be recovered from the loaded organic phase.

## 4 Conclusion

The equilibrium studies of two immiscible phases: water and tertiary amines (TDA and TOA) showed that:

TDA/kerosene and TOA/kerosene have high affinity towards HCl. Although the yield of HCl extraction by TOA is a little bit higher than that of TDA, both amines regardless their price are advisable to be used for HCl recovery from aqueous acidic solutions.

Benzene showed a positive effect on HCl extraction when it was used as diluents, but it is preferable to use kerosene +10 % (v/v) octanol-1 to have lower operation cost and lower toxicity.

The concentration of tertiary amines (TDA and TOA) and organic to aqueous phase ratio $V_{org}/V_{aq}$ have a positive effect on HCl extraction. The best operating conditions for HCl extraction are: $V_{org}/V_{aq}$ = 1.5, [Amine] = 0.5M + 10% (v/v) octanol-1, t = 40 °C.

No effect of NaCl was observed on HCl extraction and the temperature has a small negative effect on HCl extraction, the $\Delta H^o$ for the extraction reaction was found to be −4.1 kJ/mol.

Temperature and aqueous to organic phase ratio $V_{aq}/V_{org}$ have a great effect on HCl stripping from the acid loaded TDA/kerosene + 10% (v/v) octanol-1 when water was used as a stripping agent. The optimum operation conditions for HCl stripping are: $V_{H2O}/V_{org}$ = 20/1, t = 75 °C, and 3 stripping stages.

## Nomenclature

| | |
|---|---|
| M | Concentration in mol $L^{-1}$ |
| $[HCl]_i$ | Initial hydrochloric acid concentration in the aqueous phase (M) |
| $[HCl]_{aq}$ | Equilibrium hydrochloric acid concentration in the aqueous phase (M) |
| $[HCl]_{org}$ | Equilibrium hydrochloric acid concentration in the organic phase (M) |
| $[HCl]^i{}_{org}$ | Initial hydrochloric acid concentration in the organic phase (M) |
| $[TDA]_i$ | Initial tri-n-dodecylamine concentration in the organic phase (M) |
| $[TBP]_i$ | Initial tri-n-butyl phosphate concentration in the organic phase (M) |
| $[TOPO]_i$ | Initial tri-n-octyl phosphine oxide concentration in the organic phase (M) |
| $K_d$ | Distribution ratio = $[HCl]_{org}/[HCl]_{aq}$ |
| $\Delta H^o$ | Enthalpy change of the extraction reaction ($Jmol^{-1}$) |
| R | gas constant (8.314 J $K^{-1}$ $mol^{-1}$) |
| T | Thermodynamic temperature (K) |
| t | Celsius temperature (°C) |
| $V_{aq}$ | Volume of the aqueous phase |

| | |
|---|---|
| $V_{org}$ | Volume of the organic phase |
| $Y_{HCl}$ | Yield of hydrochloric acid extraction |
| $Y`_{strip}$ | Yield of hydrochloric acid stripping. |

## Acknowledgement

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