

# Isolation of ethylenediamine from industrial solution of ethylenediamine hydrochloride by electrodialysis

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**Abstract:** An industrial solution of ethylenediamine hydrochloride which contains about 18% chloride and 12% amine was electrodialysed in a five-compartment cell to obtain pure ethylenediamine. Different parameters such as alkali concentration and current density were studied to optimise conditions of maximum current efficiency for amine recovery and chloride removal from the feed solution. The Suitabilities of different ion-exchange membranes for electrodialysis were compared. Variation in current efficiency for amine recovery with increases in the concentration of amine during electrodialysis was also studied. Current efficiency for amine recovery and chloride removal is about 78% and 91% respectively under optimum conditions. The amount of electrical energy consumed is 12.1 kWh kg<sup>-1</sup> for ethylenediamine recovery and 8.6 kWh kg<sup>-1</sup> for chloride removal. The concentration of pure amine obtained after the electrodialysis was around 20%. The results are encouraging for industrial applications whereby ethylenediamines free from chlorides can be isolated.

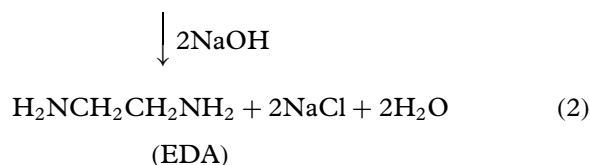
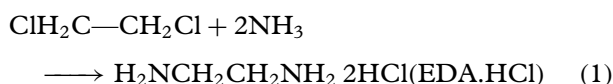
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**Keywords:** ethylenediamine; electrodialysis; ethylenediamine hydrochloride

## 1 INTRODUCTION

Electrodialysis (ED) is becoming an important pollution-free method of separation of organic amines, acids, etc from their salt solutions using ion-exchange membranes. It is widely adopted in the chemical, food and dairy industries for applications relating to separation and purification.<sup>1</sup> In addition, electrodialysis is used for the separation of salts of aliphatic acids from industrial products.<sup>1,2</sup> Many organic acids such as acetic acid,<sup>3–6</sup> citric acid,<sup>7,8</sup> dicarboxylic acids<sup>9,10</sup> and lactic acid<sup>11</sup> can be separated from their salts by electrodialysis. Strathmann *et al* have isolated dimethyl isopropyl amine from the amine sulfate.<sup>2</sup> Desalting of phenylalanine by electrodialysis has been reported recently.<sup>12</sup> Chang<sup>13</sup> *et al* has reported the conversion of ethylene amine dihydrochloride to ethylenediamine by electrodialytic water splitting.

Ethylenediamine (EDA) is an industrially important compound which is produced commercially by amination of ethylene chloride as follows:



The ethylenediamine hydrochloride solution formed by reaction (1) in the reactor comprises a mixture of amines such as triamine and tetramines in addition to the main product, EDA. The industrial solution also contains free ammonia (8.26%) and ammonium chloride (13.46%). The pure ethylenediamine is then obtained from the industrial solution by treating with sodium hydroxide as shown in reaction (2), resulting in the generation of sodium chloride which is contaminated with organic compounds and cannot be used for the generation of sodium hydroxide or other applications. The disposal of large quantities of the contaminated sodium chloride formed in the process is a threat to the environment. Though the separation of ethylenediamine has been reported to be feasible by electrodialysis,<sup>11</sup> the process has not been studied using industrial solutions. The purpose of this study therefore is to investigate the separation of pure EDA by the electrodialysis of commercial solutions of the amine hydrochloride.

The objectives of the study are to optimise the process parameters for electrodialysis using commercially available ion exchange membranes and to evaluate the product obtained in the studies.

## 2 EXPERIMENTAL

### 2.1 Electrodialysis unit

The electrodialysis unit used in the present study comprises compartments separated from each other

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by alternately placed cationic/anionic membranes. The two end compartments contained the anode and cathode and the electrolyte in the electrode compartments was fed with alkali solutions. The feed solution was the amine hydrochloride solution, supplied by a leading manufacturing company in India. The amine hydrochloride solution was fed to the central compartment of the electrodialysis unit. The compartments adjacent to the central feed compartment were fed initially with pure amine solution and pure sodium chloride solution of known composition (2% w/v). The electrodialysis unit used in the studies was fabricated to suit the requirements of the study and a schematic view of the electrodialysis unit used in the present work is shown in Fig. 1. The initial concentrations of amines and chloride in the industrial solution were 12–14% and 16–17% respectively. The end compartments containing the anode and the cathode were fed with aqueous solutions of sodium hydroxide (5–10% w/v). Deionised water was used in the preparation of solutions. During electrodialysis, the ethylenediammonium ions migrate from the central feed compartment into the adjacent product compartment through the cationic membrane and are neutralised by the  $\text{OH}^-$  ions migrating from the end compartment to yield the free amine. Similarly the  $\text{Cl}^-$  ions in the feed compartment migrate through the anionic membranes into the adjacent compartment and form NaCl. Graphite and stainless steel plates ( $1.3 \text{ dm}^2$ ) were used as anode and cathode in the end compartments.

## 2.2 Ion-exchange membranes

Choice of a suitable ion-exchange membrane is critical for electrodialysis processes as power consumption and long term operation without membrane fouling will determine the feasibility of electrodialysis processes. The ion-exchange membranes used in the present studies were purchased from M/S Ionics Corporation Inc (Ionics) and M/S Sybron Chemicals, USA (Ionac). The specifications of the membranes used in the studies are as follows.

|                   |          |         |
|-------------------|----------|---------|
| Ionics membranes: | Cationic | CR-61   |
|                   | Anionic  | AR-103  |
| Ionac membranes:  | Anionic  | MA-3475 |
|                   | Cationic | MC-3470 |

## 2.3 Analysis

Solutions in different compartments were monitored periodically for concentration changes during the course of electrodialysis. Acids and bases were determined by the usual titrimetric methods. Chloride content in the central feed compartment was estimated potentiometrically against silver nitrate solution using a silver/silver chloride working electrode and a saturated calomel electrode as reference electrode. Amine content in the EDA product solution was monitored periodically by titrating the amine solution against standard hydrochloric acid and the end point was taken after the required pH of the amine solution was attained as per the method adopted industrially. The amine content in the electrodialysed sample was also analysed by gas chromatography and the technique used permits both assay and the determination of impurities present. Water present in the amine mixture was determined by the Karl Fischer method.

## 2.4 Electrodialysis operations

Electrodialysis operations were conducted under galvanostatic conditions. Electrical power to the unit was supplied by a stabilised power supply unit (Aplab). The power supply unit was set at constant current mode and the variations in stack voltage were monitored. The current efficiency (CE) which relates the performance of the unit to a theoretical maximum is highly useful for process evaluation and was calculated using the following equation.

$$CE = \frac{FV[(c_i)_t - (c_i)_{t+\Delta t}]}{I\Delta t} \quad (3)$$

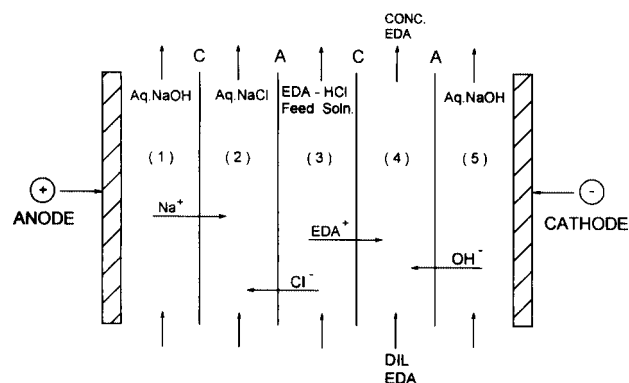
where

$F$  = Faraday constant

$I$  = Current (amps)

$V$  = Volume of solution

$(c_i)$  = Molar concentration of the ion (chloride or amine) at time  $t$  and  $t + \Delta t$ .



**Figure 1.** Typical diagram of five-compartment electrodialysis cell used in this study. A – Anion exchange membrane; C – cation exchange membrane; 1 – anode compartment; 2 – chloride ion compartment; 3 – feed amine hydrochloride compartment; 4 – pure amine compartment; 5 – cathode compartment.

## 3 RESULTS AND DISCUSSION

### 3.1 Effect of electrolyte concentration

Electrodialysis studies were conducted under galvanostatic conditions at constant current densities of  $3.0 \text{ A dm}^{-2}$  at varying concentrations of alkali in the electrode compartments. The stack voltage was lower with the Ionics membrane than with the Ionac membrane. No significant effect on current efficiencies for chloride removal and amine recovery was observed. Alkali concentrations in the anode compartment decreased during electrodialysis due to the movement of sodium ions and the discharge of hydroxyl ions

**Table 1.** Effect of electrolyte concentration on current efficiency

| Expt No | Charge passed (Ah) | Conc of electrolyte (% w/v) | Cell voltage (V) | Current efficiency (%) |                  | Conc of amine recovered (% w/v) |
|---------|--------------------|-----------------------------|------------------|------------------------|------------------|---------------------------------|
|         |                    |                             |                  | Amine recovery         | Chloride removal |                                 |
| 1       | 176                | 3                           | 12–9             | 66.0                   | 88.4             | 16.4                            |
| 2       | 277                | 5                           | 11–9             | 66.1                   | 89.6             | 15.5                            |
| 3       | 285                | 10                          | 9–7              | 64.4                   | 90.5             | 19.4                            |
| 4       | 120                | 3                           | 17–14            | 53.6                   | 82.4             | 14.5                            |
| 5       | 134                | 5                           | 14–11            | 54.6                   | 88.5             | 17.2                            |
| 6       | 174                | 10                          | 16–13            | 54.6                   | 89.6             | 16.3                            |

Expt Nos 1–3: Ionics membrane.

Expt Nos 4–6: Ionac membrane.

Compartments 1 and 5: Aq NaOH.

Compartment 2: Aq NaCl (2% w/v initial).

Compartment 3: Ethylenediamine hydro chloride feed solution.

Compartment 4: Pure ethylenediamine (2% w/v initial).

Current density:  $3.0 \text{ A dm}^{-2}$ .

at the anode. The alkali concentrations in the anode compartments have to be maintained at an optimum level of 5–10% for minimum energy consumption and operational convenience. The results are reported in Table 1.

### 3.2 Effect of current density

The current density determines the production rate and also determines the efficiency of product recovery and hence is an important parameter in electrodialysis studies. Table 2 reports the results obtained with varying current densities at constant electrolyte concentrations. It is observed that current efficiency for chloride removal decreases with increasing current densities, and for amine recovery, it increases with current density up to  $3.8 \text{ A dm}^{-2}$  and then decreases with further increases in current densities for the Ionics membrane. No such behaviour was observed for the Ionac membrane. Variation of current efficiency for

amine recovery with current density may be due to back diffusion of ethylenediamine. The concentration of the pure amine obtained in the product stream is higher than the initial feed solution (see No 4 in Table 4). The energy consumptions for amine recovery and chloride removal which depend upon stack voltage, current efficiency and product yield have been calculated and are computed to be  $12.1 \text{ kWh kg}^{-1}$  and  $8.6 \text{ kWh kg}^{-1}$  for amine recovery and chloride removal respectively under optimum conditions. The effects of current density on current efficiency during electrodialysis for amine recovery and chloride removal are shown in Fig 2.

### 3.3 Variation of current efficiency during the course of electrodialysis

Since the recovery of ethylenediamine free from chlorides is one of the main objectives of the

**Table 2.** Effect of current density on current efficiency

| Expt. No | Current density ( $\text{A dm}^{-2}$ ) | Total current passed (Ah) | Cell voltage (V) | Current efficiency (%) |                  | Conc of amine recovered (% w/v) |
|----------|----------------------------------------|---------------------------|------------------|------------------------|------------------|---------------------------------|
|          |                                        |                           |                  | Amine recovery         | Chloride removal |                                 |
| 1        | 2.3                                    | 300                       | 11.5–8.5         | 55                     | 100              | 15.1                            |
| 2        | 3.0                                    | 290                       | 11.5–9.0         | 66                     | 90               | 15.5                            |
| 3        | 3.8                                    | 233                       | 11.5–9.0         | 78                     | 91               | 20.1                            |
| 4        | 4.6                                    | 276                       | 13–10            | 62                     | 86               | 16.2                            |
| 5        | 5.5                                    | 212                       | 18–14            | 56                     | 87               | 18.5                            |
| 6        | 2.3                                    | 206                       | 11–10            | 40                     | 99.4             | 20.0                            |
| 7        | 3.0                                    | 205                       | 16–13            | 60                     | 91.0             | 18.8                            |
| 8        | 3.8                                    | 213                       | 16–13            | 60                     | 100.0            | 18.6                            |
| 9        | 4.6                                    | 175                       | 17.5–15          | 62                     | 86.0             | 20.6                            |
| 10       | 3.8                                    | 428                       | 8–7              | 80                     | 83               | 14.0                            |
| 11       | 5.6                                    | 300                       | 10–9             | 74                     | 82               | 13.5                            |

Expts 1–5: Ionics membrane (5% NaOH).

Expts 6–9: Ionac membrane (10% NaOH).

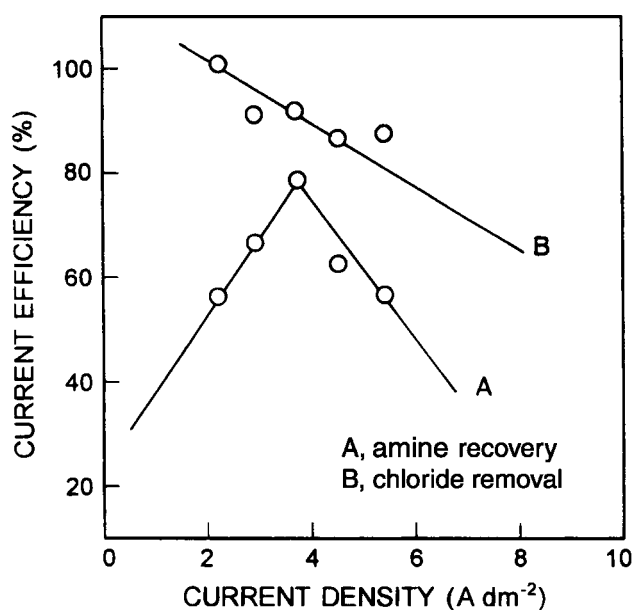
Compartments 1 and 5: Aq NaOH.

Compartment 2: Aq NaCl (2% w/v).

Compartment 3: Ethylenediamine hydrochloride feed solution.

Compartment 4: Pure ethylenediamine (2% w/v initial).

Conditions: Solutions in different compartments as in Table 1.



**Figure 2.** Typical plot showing the effect of current density on current efficiency during electro dialysis of ethylenediamine hydrochloride.

present study, the variations in amine concentration and current efficiency were computed during the course of electro dialysis. The results are reported in Table 3. It is seen that the current efficiency decreases during electro dialysis and there is a fall in current efficiency with increases in the amine concentration in the product compartment. The decrease may be due to the reverse diffusional transport of the neutral ethylenediamine which increases with amine concentration and is also higher at low current densities. The decrease in current efficiency may also be due to the fouling of the membrane by adsorption of the organic compound at higher concentrations of the amine.<sup>14</sup> Hence an optimum concentration of the recovered amine has to be established.

### 3.4 Product evaluation

After the completion of an electro dialysis batch, the amine solution obtained was evaluated by comparing it with the analysis of the ethylenediamine hydrochloride solution used as a feed solution in the process. Table 4 gives the physical and chemical characteristics of the industrial feed solution and the product isolated by electro dialysis. It is seen that the products obtained in the electro dialysis trials (Expts 1–6) have chloride contents ranging from 0.15 to 0.9% while the chloride content in the feed solution was 18.3%. The amine content in the feed solution was 12% compared with an amine content of 13.6–18.2% in the products as obtained in the electro dialysis trials. The electro dialysis products however had a higher pH value than the feed solution and the increase in alkalinity is due to the migration of alkali from the end compartment. The sodium ion content of the product solution was also around 1.37% and is due to the leakage of sodium ions from the end compartment but this can be minimised in the pilot-scale operation with the use of multiple repeating units. The electro dialysed solution was analysed by the manufacturing company and it was observed that the composition of the amine was unchanged in the electro dialysis product which can be subjected to normal fractionation to separate the pure ethylenediamine. Electro dialysis avoids the need for alkali treatment, evaporation and effluent disposal steps. Another advantage is that the pure sodium chloride isolated in the electro dialysis process can be used for regeneration of sodium hydroxide, depending on the economics of the process.

### 3.5 Fixing the chloride as ammonium chloride

Studies were conducted to fix the chloride ions as ammonium chloride by using ammonium salts as electrolytes in the end compartments. Ammonium carbonate (10%) was used as the anolyte and a mixture of 10% ammonium hydroxide and 20% ammonium sulfate was used as catholyte and electro dialysis

**Table 3.** Effect of concentration of amine in the output compartment (compartment 4) on current efficiency during electro dialysis

| Expt No | Current density (A dm <sup>-2</sup> ) | Conc of amine in compartment 3 (% w/v) | Conc of amine recovered in compartment 4 (% w/v) | CE for amine recovery (%) |
|---------|---------------------------------------|----------------------------------------|--------------------------------------------------|---------------------------|
| 1a      | 2.3                                   | 14.5                                   | 12.2                                             | 52.7                      |
| 1b      |                                       | 15.2                                   | 19.9                                             | 26.3                      |
| 2a      | 3.0                                   | 16.0                                   | 7.9                                              | 100                       |
| 2b      |                                       | 14.0                                   | 13.8                                             | 46.9                      |
| 3a      | 3.8                                   | 12.9                                   | 8.3                                              | 80.7                      |
| 3b      |                                       | 15.4                                   | 15.9                                             | 41.7                      |
| 4a      | 4.6                                   | 18.7                                   | 8.1                                              | 74.3                      |
| 4b      |                                       | 19.7                                   | 19.4                                             | 44.7                      |
| 5a      | 4.6                                   | 13.8                                   | 6.4                                              | 100                       |
| 5b      |                                       | 16.5                                   | 16.2                                             | 43.6                      |

Note:

(1) Expt Nos 1a, 2a, 3a, 4a and 5a indicate the results obtained during the initial period of electro dialysis when the current efficiency is high.

(2) Expt Nos 1b, 2b, 3b, 4b and 5b indicate the results obtained towards the end of electro dialysis when there is a decrease in current efficiency.

**Table 4.** Analytical data of some solutions of ethylenediamine solution obtained by electrodialysis and feed ethylenediamine hydrochloride solution

| Parameter                | Feed solution          | Experiments         |                     |                     |                     |                     |                     |
|--------------------------|------------------------|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|
|                          |                        | 1                   | 2                   | 3                   | 4                   | 5                   | 6                   |
| Appearance               | Yellowish clear liquid | Light yellow liquid | Light yellow liquid | Light yellow liquid | Light yellow liquid | Light yellow liquid | Light yellow liquid |
| Density                  | 1.046                  | 1.016               | 1.038               | 1.02                | 1.02                | 1.022               | 1.027               |
| pH                       | 9.60                   | 12.9                | 13.69               | 13.62               | 13.12               | 13.75               | 12.37               |
| Amines (wt %)            | 12.0                   | 15.12               | 13.6                | 15.0                | 18.2                | 16.3                | 16.4                |
| Ammonia (wt %)           | 8.26                   | 0.2                 | 0.8                 | 0.6                 | 0.7                 | 0.8                 | 0.52                |
| Ammonium chloride (wt %) | 13.46                  | Nil                 | Nil                 | Nil                 | Nil                 | Nil                 | Nil                 |
| Chloride (wt %)          | 18.3                   | 0.9                 | 0.6                 | 0.3                 | 0.15                | 0.15                | 0.06                |
| NaOH (wt %)              | Nil                    | Nil                 | 2.1                 | 2.8                 | 1.7                 | 3.0                 | 1.37                |

proceeded with a current efficiency of 27% for amine recovery and 80% for chloride removal. It was observed that the amine concentration can be built up to a level of 12% with ammonia-based electrolytes. However, ammonium hydroxide solutions are not suitable for pilot-scale operations as electrolytes due to higher stack voltages and loss of ammonia during electrodialysis.

### 3.6 Electrodialysis with sodium carbonate as electrolyte

Studies were conducted with the use of sodium carbonate solutions as anolyte and catholyte as cost effective alternatives to sodium hydroxide and the results (Table 5) show that the current efficiency for amine recovery and chloride removal were similar to the values obtained with sodium hydroxide. But carbonate ion migration to the amine compartment resulted in higher values for amine and the stack voltages were also higher, resulting in higher energy consumption ( $14.2 \text{ kWh kg}^{-1}$  vs  $9.5 \text{ kWh kg}^{-1}$ ) when using sodium carbonate as the electrolyte than when using sodium hydroxide as the electrolyte. Comparative values of energy consumption and current

efficiency values obtained for different electrolytes have been summarised in Table 5.

## 4 CONCLUSIONS

Ethylenediamines can be separated in pure form from industrial solutions of ethylenediamine hydrochloride by electrodialysis. Concentration of the recovered amine can be maintained equal to or marginally higher than the feed concentrations. Under optimum conditions, the current efficiencies for amine recovery and chloride removal are 78–81% and 83–91% respectively. Chloride concentrations in EDA can be brought down to 0.06%. Energy consumption for amine recovery is  $9.5 \text{ kWh kg}^{-1}$  under optimum conditions. Chloride ions can be fixed as sodium chloride or ammonium chloride but recovery as sodium chloride is more energy efficient. Sodium carbonate can also be used as an electrolyte instead of sodium hydroxide. The electrodialysis unit is amenable for scale-up and electrodialysis can be developed as a viable industrial process for the separation of pure ethylenediamine from the amine hydrochloride. Further studies on a pilot scale are to be performed.

**Table 5.** Effect of various types of electrolytes

| Expt No | Anolyte                                                       | Catholyte                        | Charge passed (Ah) | Current efficiency (%) |                  | Energy consumption for amine ( $\text{kWh kg}^{-1}$ ) |
|---------|---------------------------------------------------------------|----------------------------------|--------------------|------------------------|------------------|-------------------------------------------------------|
|         |                                                               |                                  |                    | Amine recovery         | Chloride removal |                                                       |
| 1       | 10% NaOH                                                      | 10% NaOH                         | 366                | 79.7                   | 82.4             | 9.5                                                   |
| 2       | 10% $\text{Na}_2\text{CO}_3$                                  | 10% $\text{Na}_2\text{CO}_3$     | 390                | 82.1                   | 89.4             | 14.2                                                  |
| 3       | 10% $\text{Na}_2\text{CO}_3$                                  | 10% NaOH                         | 218                | 77.5                   | 92.5             | 11.0                                                  |
| 4       | 10% $\text{NH}_4\text{OH}$ + 20% $(\text{NH}_4)_2\text{CO}_3$ | 10% $(\text{NH}_4)_2\text{CO}_3$ | 62                 | 19.2                   | 83.1             | —                                                     |

Cell voltage = 8.6V, 13.6V, 10.0V and 16–23V respectively for expts 1–4.  
Current density =  $3.7 \text{ A dm}^{-2}$  for expts 1–3 and  $2.0 \text{ A dm}^{-2}$  for expt 4.

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