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Recovery of ammonia/ammonium sulphate from synthetically prepared effluent streams

K. Sarangi, N.C. Rout, T. Subbaiah, S. Anand*, R.P. Das

Hydro and Electro Metallurgy Departments, Regional Research Laboratory (CSIR),
Bhubaneswar – 751 013, Orissa, India

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ABSTRACT

Oxidative ammonia leaching of sulphidic ores and reductive leaching of oxidic ores with sulphur based reductants in ammoniacal medium produce effluent streams containing ammonium sulphate. The various options for treating such solutions are: (a) concentration of ammonium sulphate by reverse osmosis or electro-dialysis (b) electrolytic decomposition of ammonium sulphate for ammonia and sulphuric acid recovery and (c) lime boil for ammonia recovery. Experimental data generated for concentrating synthetically prepared ammonium sulphate by reverse osmosis and by electro-dialysis gave maximum concentrations of ~40 and 270g/l respectively. Electro-decomposition studies showed that current efficiency depended on various factors such as initial ammonium sulphate concentration, current density and presence of metal ion impurities. A maximum of 87% current efficiency was achieved with initial ammonium sulphate concentration > 70g/l while keeping the current density as 100mA/cm^2. The energy requirement during electro-decomposition of ammonium sulphate for producing ammonia was found to be less as compared to other processes used for its production. Ammonia recovery through lime boil gave ~93% efficiency but the process suffers due to disposal problem of calcium sulphate. A combination of electrochemical decomposition and electro-dialysis operations has been suggested as a viable approach for ammonia and sulphuric acid recovery. © 2004 SDU. All rights reserved.

Keywords: Electro-dialysis; Electrodecomposition; Ammonia; Ammonium sulphate

1. INTRODUCTION

Ammonia leaching is extensively used for recovery of metal values from sulphide and oxide ores. During oxidative leaching of sulphides, most of the sulphur gets converted to ammonium sulphate as in the Sherrit Gordon Process for nickel and cobalt recovery. In case of oxidic ores ammonium sulphate is generated when sulphur based reductants are used during leaching in ammoniacal medium. Depending on the process steps there may be various streams containing ammonium sulphate of varying concentrations. Treatment of manganese nodule with sulphur based reductants such as sulphur dioxide, ferrous sulphate, thiosulphate etc., would also produce ammonium sulphate during leaching (Anand *et al.*, 1988; Acharya *et al.*, 1989; Niinae *et al.*, 1996; Das and Anand 1997). In such processes free ammonia will be recovered and recycled but ammonium sulphate would remain in the effluent streams. The recovery/utilization of ammonium sulphate is important both from the environmental and economics point of view. The approach towards treatment of dilute ammonium sulphate solutions should be to (i) increase the salt concentration to a maximum value or (ii) decompose the ammonium sulphate for recovery of ammonia for recycling and (iii) to generate minimum solid/liquid effluents while processing such streams. One of the techniques which may be employed is reverse osmosis (RO). This is widely used for desalination of water (Metcalf and Hunt, 1989) and separation and concentration of heavy metals (Hanra and Ramchandhran, 1996) and is regarded as a cheap method for concentrating solute concentrations in solutions. Similarly electro-dialysis/electro-electrodialysis is another technique for concentrating/ separating acid and salt solutions or various cations and anions (Cherif *et al.*, 1988; Norton and Buehler 1994; Pfromm, 1997; Rockstraw and Scamehoron, 1997; Shah *et al.*, 1999; Luo and Wu, 2000). Electrolytic decomposition of salt could be another option as it has been investigated for sodium sulphate (Jorissen and Simmrock, 1991; Jorissen and Simmrock, 1996). The other method of treating such solutions could be lime boil for ammonia recovery.

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* Corresponding author. E-mail: s_p_anand@hotmail.com

However, these options have never been tested for treatment of dilute ammonium sulphate solutions. Due to lack of information/data available in the literature on these aspects, the present investigation was taken up with the pure ammonium sulphate solutions.

2. EXPERIMENTAL

2.1. Reverse osmosis (RO)

The reverse osmosis unit along with spirally wound thin film composite membranes used in the present work were supplied by Permionics, Vadodara, India. The module size was 6.35cmx13.6cm. The effective membrane area of the RO module was $0.72m^2$. For reverse osmosis, one feed pump (Aroma Legend LS –75 model)) was used for pumping the feed solution and one high pressure pump (Prakash FIG PIRP-3 model) was used to pass the solution through the RO membrane.

To increase the concentration of $(NH_4)_2SO_4$ by Reverse Osmosis (RO) technology, $(NH_4)_2SO_4$ concentration in feed solution was varied from 1 to 20g/l. Twenty litres of solution was taken in a bucket for each experiment. This solution was pumped from the bucket by a feed pump and was passed through the system by a high pressure RO pump. The pressure regulating valve connected to the system was suitably adjusted to set the upstream pressure at the desired value. The reject stream was taken back to the feed bucket. The samples were withdrawn from the diluate (the initial dilute stream is termed as diluate) and concentrate streams and were analysed for ammonium sulphate concentration.

2.2. Electro-dialysis

For electro-dialysis work, thin film of styrene-divinyl benzene co-polymer was prepared by suitable plastic processing technique at Central Salt and Marine Chemicals Research Institute, Bhavnagar, India. Thin film was then converted to anion exchange membrane by chloro-methylation-ammination and to cation exchange membrane by sulphonation process. Seven pairs of cation and anion exchange membranes were packed alternately in between two electrodes. Cation and anion exchange membranes were packed in a PVC housing. This served the purpose of holding the electrodes in place, provided the inlet connections for feeding separately two streams (diluted and concentrate stream) at the bottom of the stack and outlet connections for treated and concentrated stream from the top of the stack. The cathode of S.S. 316 plate (~2mm thick) and titanium anode of expanded metal type were used. The spacer gaskets used for separating the two adjacent membranes were of polyethylene net having more than 80% opening. In electro-dialysis process three numbers of PP pumps (Special Devices MDP 20L/h model) were used to circulate feed, concentrate and electrode wash solution.

The feed solution containing $(NH_4)_2SO_4$ was split into two streams and fed into alternate set of compartments in the stack with the help of two peristaltic pumps. Direct current was passed through this unit and the ions in solution migrated to electrodes with opposite polarity, but due to the membrane barriers, one set of alternate chambers in the stack get depleted in $(NH_4)_2SO_4$ content, while the other remaining chambers get concentrated with $(NH_4)_2SO_4$. The samples from the concentrate and diluate stream were drawn out continuously from the system for analysis. The conductivity of samples was also measured. The chambers adjacent to the electrodes were continuously flushed with dilute Na_2SO_4 solution. For concentration of $(NH_4)_2SO_4$ through electro-dialysis, the initial concentration of $(NH_4)_2SO_4$ in feed was taken as 110g/l. The other chemicals such as sodium hydroxide and sulphuric acid used were of A.R. grade. Analysis of $(NH_4)_2SO_4$ was carried out titrimetrically using methyl red indicator and the conductivity of the solution was measured using a conductivity meter (Systronics conductivity meter Model 304).

2.3. Electrolytic decomposition of ammonium sulphate

Five hundred millilitres corning beaker separated by polypropylene diaphragm was used as an electrolytic cell. The anode was a titanium substrate insoluble material of 0.3cm thick having dimensions as: 10cm length and 5cm width. The cathode was a stainless steel sheet having the dimensions as: 10cm length and 5cm width. Fresh electrodes were used for each experiment. The cathodes were carefully polished with fine (600 grade) emery paper, washed under running tap water, scrubbed with filter paper, rinsed with distilled water, and finally air dried. The electrodes were placed into the cell and connected to the circuit for ammonia production. The electrolysis was carried out by applying DC voltage from a regulated power supply unit and the current was controlled by a rectifier. The cell voltage was measured by connecting a voltmeter to the anode and the cathode in two compartment cell. The cathodic current efficiency was measured by analyzing the ammonia formed after electrolysis at the cathode compartment.

2.4. Lime boil for decomposition of ammonium sulphate

During these experiments solution containing 110g/l ammonium sulphate was heated in a closed reactor with weighed quantity of lime at desired temperatures for specific period. The contents were cooled, filtered and ammonia was estimated volumetrically in the filtered solution to determine the percentage of ammonium sulphate decomposition.

3. RESULTS AND DISCUSSION

3.1. Reverse osmosis (RO) experiments

3.1.1. Effect of $(NH_4)_2SO_4$ concentration on solute separation

Ammonium sulphate concentration in feed solution was varied from 1-20g/l while keeping the pressure constant at 13.6kg/cm^2. The solute separation of the membrane at different ammonium sulphate concentrations in feed are shown in Fig. 1. It is observed that the solute separation decreased from 99.6 to 94.2% with increase of ammonium sulphate concentration from 1 to 20g/l. With this experimental set up and membrane, ammonium sulphate could be enriched up to 40g/l from an initial concentration of feed solution as 1g/l. Further enrichment was not possible which could be due to increase of solution viscosity.

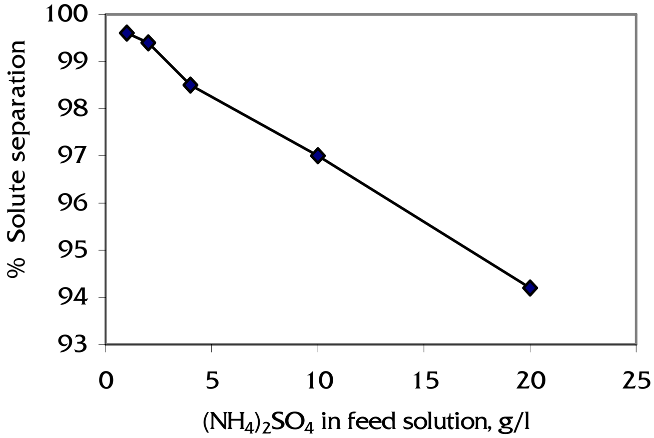

Figure 1. Effect of $(NH_4)_2SO_4$ concentration on % solute $(NH_4)_2SO_4$ separation

3.1.2. Effect of pressure on solute separation

The solute separation at different pressure was studied in the range of 5.45 to 17kg/cm^2 and is given in Fig. 2.

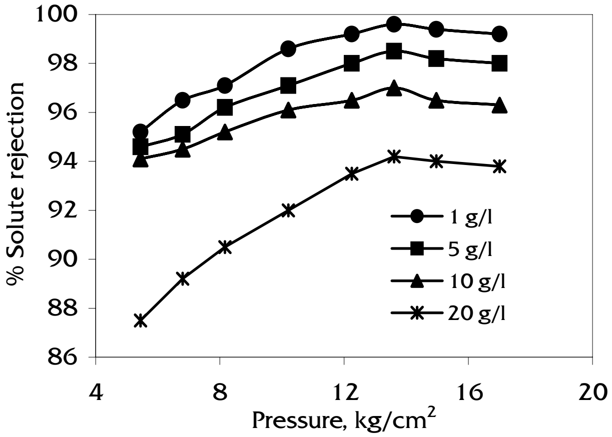

Figure 2. Effect of pressure on solute $(NH_4)_2SO_4$ separation

This study was carried out with different $(NH_4)_2SO_4$ concentrations in feed solution. It was observed that the solute separation increased with increase of pressure from 5.45 to 13.6kg/cm^2. The maximum solute separations with 1g/l and 20g/l $(NH_4)_2SO_4$ in feed solution were 99.6% and 94.2% respectively at 13.6kg/cm^2. So in a continuous enrichment process of $(NH_4)_2SO_4$ from 1g/l to 40g/l, the solute separation would decrease from 99.6 to 94.2%.

3.2. Results on electro-dialysis (ED) for concentration of $(NH_4)_2SO_4$

The mechanism of concentrating $(NH_4)_2SO_4$ through electro-dialysis process is shown in Fig. 3. The initial concentration taken in the feed solution was 110g/l with conductivity of 67.7ohm^{-1}. Table 1 shows the variation of current and conductivity with time for a typical experiment. Fig. 4 shows that ammonium sulphate could be concentrated from 110 to 268g/l. It was observed that due to transport of ~30% water molecules, input and output concentrations of ammonium sulphate became almost constant at ~270g/l, hence further enrichment was difficult. Though in this typical experiment the initial concentration was taken as 110g/l, but it is possible to achieve a concentration of ~270g/l even with low initial concentrations of ammonium sulphate e.g. in case of very dilute streams, the solution can be concentrated upto 40g/l by RO followed by further concentration by ED. The energy consumed for concentrating $(NH_4)_2SO_4$ was calculated to be 0.315 to 0.4kWh/kg of $(NH_4)_2SO_4$.

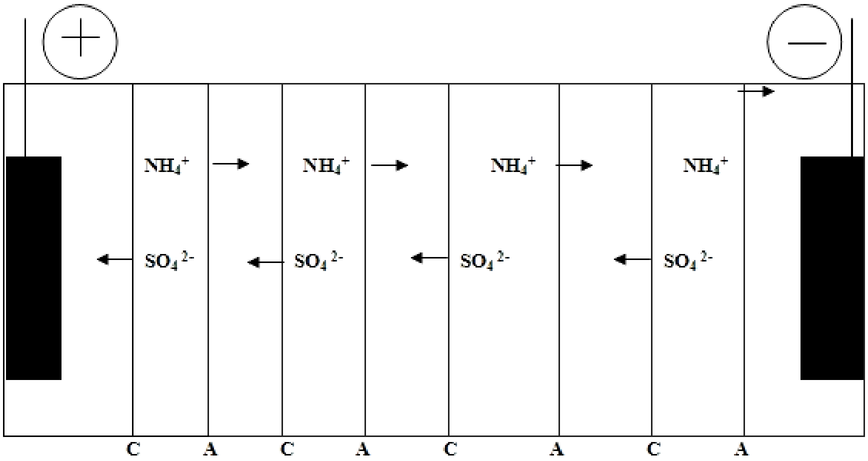

Figure 3. Separation of ions (NH_4^+ and SO_4^{2-}) in multi-compartment electrolytic cell. A = anion exchange membrane, C = cation exchange membrane

Table 1
Variation of current with time

Time, min	Current (A)	Conductivity, ohm^{-1}		
		Original	Diluate	Concentrate
0	0.85	67.7	-	-
10	0.95	-	65.1	68.1
20	0.99	-	64.3	68.4
35	1.0	-	62.8	70.6
50	0.99	-	58.3	71.8
60	0.99	-	55.5	73.4
75	0.98	-	53.8	75.8
85	0.98	-	51.7	74.6
95	0.97	-	48.1	75.2
105	0.96	-	44.2	75.2
120	0.89	-	31.8	75.4
140	0.82	-	25.3	75.8
160	0.77	-	20.4	75.9
165	0.73	-	18.1	76.1

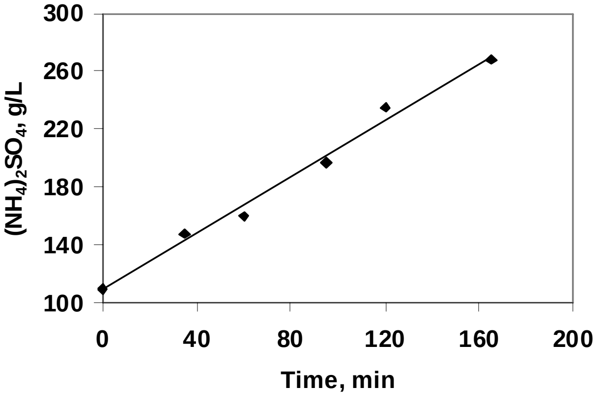

Figure 4. Effect of time on ammonium sulphate concentration during electro-dialysis

3.3. Electro-decomposition of ammonium sulphate

While both reverse osmosis and electro-dialysis processes result in enrichment of amminium sulpahte, electrolytic decomposition of ammonium sulphate is for the separation of ammonia and sulphuric acid as shown by the following chemical reaction:

$(NH_4)_2SO_4 \rightarrow 2\ NH_3 + H_2SO_4$ (1)

The electro-decomposition experiments were carried out at different current densities, ammonium sulphate concentrations and time intervals. Since small amounts of metal ion impurities will generally be associated with the effluent streams, effect of presence of these impurities was also studied.

3.3.1. Effect of current density

Current density was varied from 50 to 1000A/m^2. The results given in Table 2 show that with the increase in current density, cell voltage increases resulting in enhancement of energy consumption. Highest current efficiency of 87% was observed by maintaining current density of 100A/m^2. Decrease in current efficiency above current density of 100A/m^2 has also been reported during electo-deposition of cobalt from cobalt sulphate bath in the presence/absence of boric acid (Das and Subbaiah, 1984; 1987). This was attributed to the inadequate supply of charged ions to the interface resulting in hydrogen evolution reaction.

Table 2
Effect of current density on decomposition of $(NH_4)_2SO_4$. Conditions: catholyte 100g/l $(NH_4)_2SO_4$, anolyte 10g/l. H_2SO_4, duration 2.5h, temp. ambient

Sl.No.	Current Density A/m^2	Catholyte end pH	% current efficiency	Cell voltage volts	Depletion of $(NH_4)_2SO_4$ g/l
1	50	8.4	55.13	2.3	4.47
2	100	8.7	87.02	2.6	10.05
3	200	9.2	73.41	3.1	17.85
4	400	9.7	41.32	3.8	25.67
5	1000	10.3	32.22	4.9	36.83

3.3.2. Effect of catholyte concentration

The concentration of catholyte was varied from 25 to 100g/l while concentration in the anolyte was kept at 10g/l sulphuric acid. It was observed that at ammonium sulphate concentration of 100g/l in the catholyte, higher current efficiency and lower cell voltage were obtained whereas at a concentration of 50-70g/l low current efficiency was recorded. The results of these studies are shown in Table 3.

3.3.3. Effect of time on electro-decomposition of ammonium sulphate of different initial concentrations

Effect of time on electro-decomposition of ammonium sulphate at different initial concentrations is shown in Fig. 5. With the increase in time, the depletion increases for all the concentrations taken up in the present study.

Table 3
Effect of ammonium sulphate concentration in catholyte. Conditions: duration 2.5h, current density 200A/m^2, temp. ambient

Sl No.	Anolyte H_2SO_4 g/l	Catholyte $(NH_4)_2SO_4$ g/l	Current Efficiency %	Cell voltage V	Depletion of $(NH_4)_2SO_4$ g/l
1	10	24.55	25.13	3.4	5.58
2	10	42.42	30.15	3.2	6.70
3	10	61.39	40.20	2.9	8.93
4	10	81.47	50.25	2.9	11.16
5	10	102.68	73.41	2.9	16.30

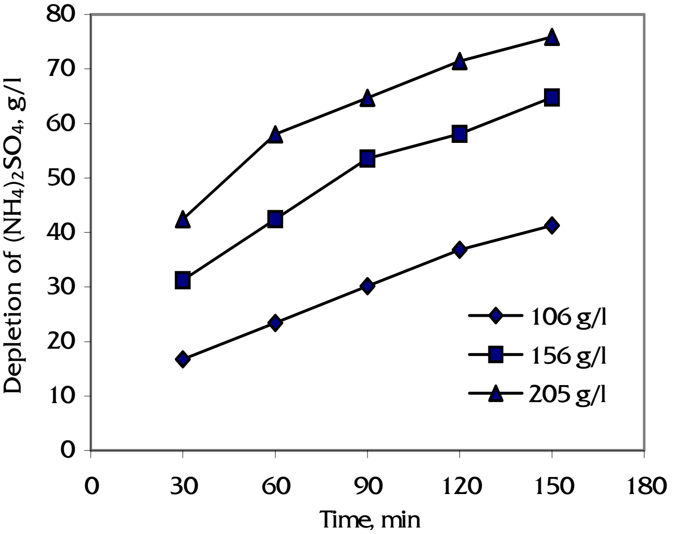

Figure 5. Effect of time on depletion of ammonium sulphate of various concentrations

3.3.4 Effect of addition of metal ion impurities

Effect of addition of Cu(II), Ni(II) and Co(II) on electro-decomposition of ammonium sulphate is shown in Table 4. The addition of metal ions results in decrease in current efficiency. Similar results were obtained by Gogia and Das (1991) during electro-winning of nickel in the presence of Co(II), Cu(II), Fe(II) and Fe(III) from nickel sulphate bath containing boric acid and sodium sulphate. This observation warrants that the metal ion impurities be removed to a minimum level prior to subjecting the ammonium sulphate solution to electrolytic decomposition.

Table 4
Effect of presence of impurities on decomposition of $(NH_4)_2SO_4$. Conditions: anolyte 10g/l. H_2SO_4, duration 2.5h, current density 200A/m^2, temp. ambient

Sl No.	Catholyte $(NH_4)_2SO_4$ g/l	M*SO_4 in catholyte g/l	Current efficiency %	Cell voltage V	Final pH of the catholyte
1	100	0	63.97	3.0	8.59
2 (Cu)	100	0.56	54.84	3.2	8.86
3 (Ni)	100	0.56	55.54	3.2	9.21
4 (Co)	100	0.56	60.62	3.1	8.57

* M is Cu(II), Ni(II) or Co(II)

The studies indicate that the electro-chemical splitting technique is a viable process to simultaneously generate ammonium hydroxide and sulphuric acid. The presence of small quantities of metal ions such as Cu(II), Ni(II) and Co(II) appear to decrease the current efficiency. The energy required for production of ammonia from the effluent streams containing ammonium sulphate need to be comparable with other processes for ammonia production. Such a comparison is made in Table 5 (Kirk-Othmer, 1978). It can be inferred that ammonia produced through electro-decomposition would require minimum energy.

Table 5
Comparison of energy requirements for NH_3 production. (Kirk-Othmer, 1978).

Process	Requirement, kWh/kg
Natural gas reforming	10.34
Naphtha reforming	10.98
Fuel oil partial oxidation	11.31
Coal gasification	
Lurgi	14.54
Koppers-Totzek	16.18
Electrochemical splitting method	8.0

3.4. Results on lime boil for decomposition of ammonium sulphate

Recovery of ammonia through lime boil could provide another option for treatment of ammonium sulphate containing solutions. In this process ammonia is generated and the sulphate is rejected as calcium sulphate. The decomposition reaction will be as given below:

$$(NH_4)_2SO_4 + CaO \rightarrow CaSO_4 + 2NH_3 + H_2O \qquad (2)$$

The results obtained on varying the process parameters such as time, temperature, and amount of lime are discussed in the following sections.

3.4.1. Effect of amount of lime

Effect of amount of lime (4g to 12g/100ml of solution) was studied at 100°C and decomposition time was kept as 1h. The results given in Fig. 6 show that with the increase of CaO upto 10g/100ml of solution, % decomposition of ammonium sulphate increased and further increase in amount of CaO did not have any significant effect.

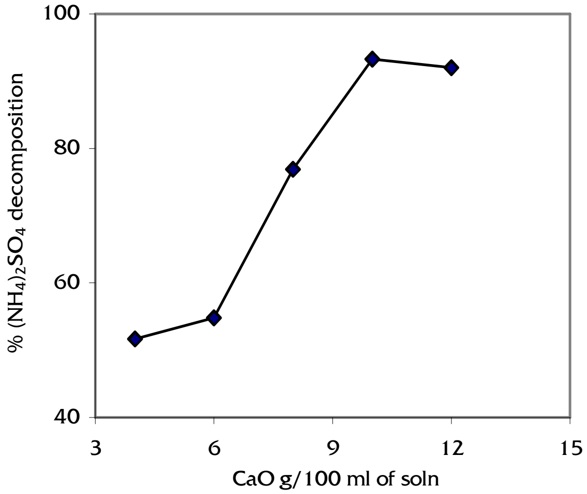

Figure 6. Effect of amount of lime on % decomposition of ammonium sulphate Conditions: $(NH_4)_2SO_4$ 110g/l, time 60min, temp. 100°C

3.4.2. Effect of time

In this set of experiments the amount of lime was kept fixed as 10g CaO/100ml ammonium sulphate solution and decomposition temperature was maintained as 100°C while the time was varied between 30 and 60min. The results given in Fig. 7 show that with the increase in time from 30 to 50min, the % decomposition of ammonia sulphate increased from 83 to 93% and further increase in time had no effect.

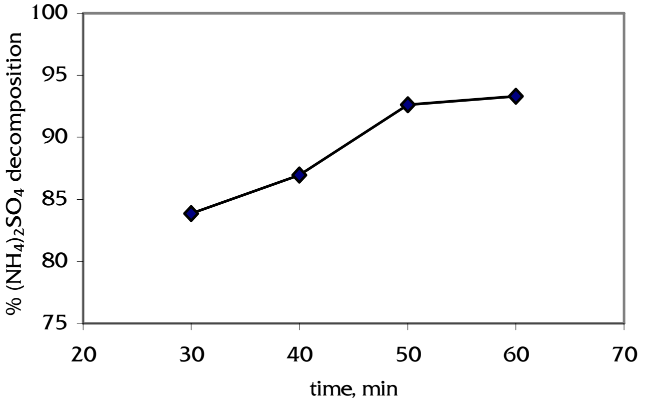

Figure 7. Effect of time on % decomposition of ammonium sulphate. Conditions: $(NH_4)_2SO_4$ 110g/l, CaO 10g/100ml soln, temp. 100°C

3.4.3. Effect of temperature

Effect of temperature was studied in the range of 60 to 110°C while keeping other two parameters fixed at 10g CaO/100ml of ammonium sulphate solution and time as 60min. The % decomposition of ammonium sulphate increased upto 90°C and further increase in temperature had no effect (Fig. 8). A maximum of 93% decomposition could be achieved.

The studies on decomposition of ammonium sulphate by lime boil have revealed that ~93% of $(NH_4)_2SO_4$ can be decomposed to recover ammonia. This option does recover ammonia for recycling back to the system but the sulphate need to be discarded as gypsum.

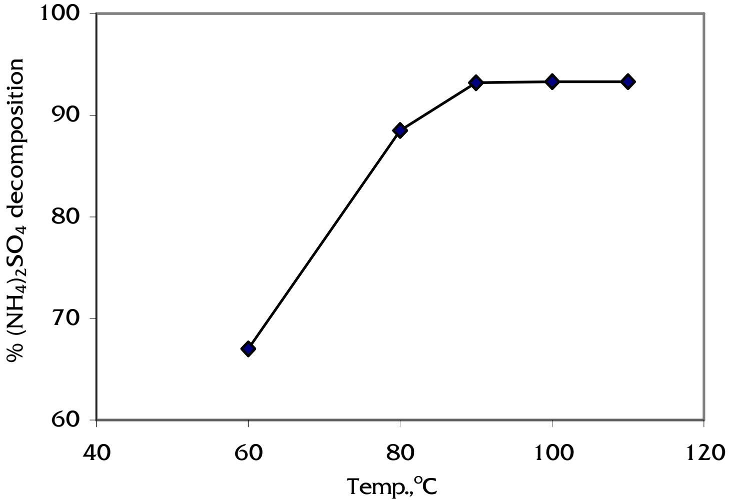

Figure 8. Effect of temperature on % decomposition of ammonium sulphate. Conditions: $(NH_4)_2SO_4$ 110g/l, CaO 10g/100ml soln., time 60min.

From the above studies, it may be concluded that for streams having low ammonium sulphate content (1-20g/l), the concentration can be improved by reverse osmosis. This may also be achieved by electro-dialysis but during electro-dialysis energy is consumed whereas the reverse osmosis provides a comparatively cheaper option. The dilute effluent stream can first be subjected to reverse osmosis followed by further concentration through electro-dialysis. Crystallization of the concentrated solution will give ammonium sulphate. In this option ammonium sulphate is recovered as a by-product resulting in consumption of ammonia/ sulphur based reductants as both these are not regenerated for recycling back to the process.

Decomposition of ammonium sulphate by lime boil is quite efficient as 93% decomposition takes place and ammonia is regenerated for recycling. To achieve such high efficiency, the requirement of lime is 2.5 times stoichiometric (Fig. 6). This consumption can be reduced by carrying out decomposition countercurrently. Even with stoichiometric consumption of lime, ~4 tons gypsum (dry basis)/ton ammonia will be produced. Disposal of such large tonnage of gypsum puts this option at a disadvantageous position.

It seems that a combination of electro-decomposition and electro-dialysis is a viable approach as per the conceptual flow sheet (Fig. 9) to recover ammonia and sulphuric acid from ammonium sulphate.

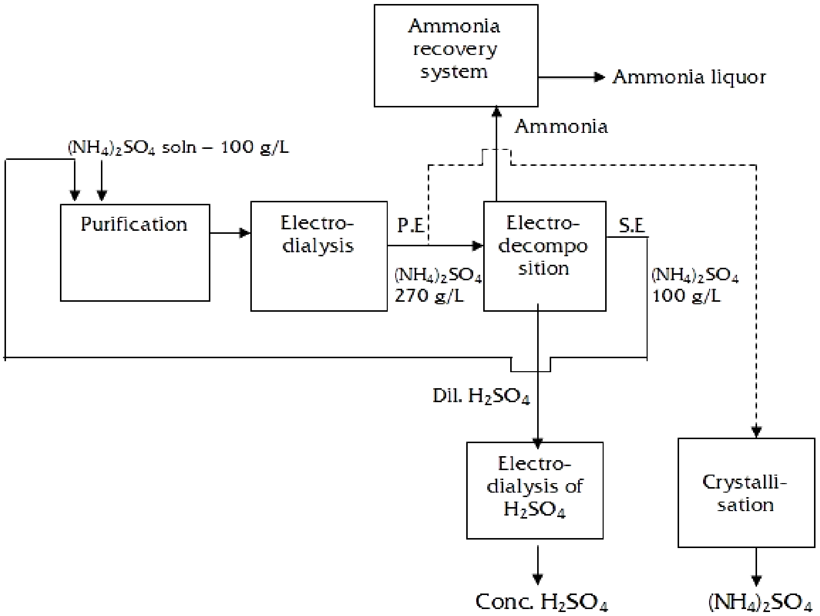

Figure 9. Conceptual flow-sheet for production of NH_3 and sulphuric acid by a combination of electro-dialysis and electro-decomposition techniques (----- lines indicate the option for crystalization of ammonium sulphate)

In case of electro-decomposition, it is observed that the energy requirement for electrochemical splitting is less when compared to the energy required for producing ammonia using different methods. By recycling the anolyte and using electro-dialysis technique, the concentration of sulphuric acid in the anolyte can be increased and theoretically 2.88 tons of sulphuric acid can be produced for each ton of ammonia. This method also avoids the problem of disposing calcium sulphate.

4. CONCLUSIONS

Very dilute solutions of $(NH_4)_2SO_4$ (1 to 20g/l) can be concentrated through reverse osmosis to obtain ~40g/l concentration. These solutions can be concentrated up to ~270g/l using electro-dialysis. Water transport along with $(NH_4)_2SO_4$ limits further up-gradation of $(NH_4)_2SO_4$ in solution. Energy consumption in the process is calculated to be 0.315 to 0.4KWh/kg of $(NH_4)_2SO_4$ concentrated. Ammonia recovery by decomposition of $(NH_4)_2SO_4$ by lime boil gives an efficiency of ~93% but gypsum which is produced as a solid effluent puts the process at a disadvantageous position. Electro-decomposition is an efficient process for recovery of ammonia and sulphuric acid. A comparison made on the energy requirement for producing ammonia using different methods revealed that electro-decomposition would require less energy as compared to that required by other processes such as natural gas reforming, naptha reforming, fuel oil partial oxidation and coal gasification. This process does not generate any solid or liquid streams which need to be disposed hence it is an environment friendly option. A combination of electrochemical splitting and electro-dialysis operations is a viable option for treating $(NH_4)_2SO_4$ solutions as proposed in the conceptual flow sheet.

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