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**"A PROCESS FOR THE PREPARATION OF SOLID AMMONIUM ~~KERENIX~~
KAXE PERSULFATE"**

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, Rafi Marg,
New Delhi-1, India, an Indian registered body incorporated under
the registration of Societies Act (Act XXI of 1860).

The following specification describes the nature
of this invention.

This is an invention by Mahadev Govind POTDAR, Scientist,
Rengachari SRINIVASAN, Scientist and Handady Venkatakrishna UDUPA,
Director, All of Central Electrochemical Research Institute,
Karaikudi-623 006, Tamil Nadu- all Indian citizens.

This invention relates to a process for the preparation of solid ammonium persulfate.

Hitherto solid ammonium persulfate has been prepared by electrolyzing ammonium sulfate, sulfuric acid solution and concentrating and crystallising the resultant solution or cool the electrolyte separates down to 10-15°C in order to get solid ammonium persulfate. The composition of the electrolyte in vogue does not yield solid ammonium persulfate during electrolysis.

This is open to the objection that the unit operation of heating during concentration of the solution tends to decompose the heat sensitive persulfate in one process and the other process of refrigeration calls for additional cooling step. Also the refrigerated product contains 5-10% $(\text{NH}_4)_2 \text{SO}_4$ as impurity.

The object of this invention is to obviate the above mentioned difficulties by developing a process for making solid ammonium persulfate directly from an electrolytic cell and with a higher purity than is obtainable by chilling the electrolyte in the known process.

To these ends the invention broadly consists in preparation of aqueous solution of ammonium sulfate and sulfuric acid, electrolyzing between platinum and lead electrodes in a suitable electrolyzing cell at optimum conditions of concentration, temperature, current volume and flow rate so as to get solid ammonium persulfate directly from the electrolyser in the form of slurry which is processed further for getting dry, crystalline powder of ammonium persulfate ready for market.

The composition of electrolyte i.e., ammonium sulfate and sulfuric acid is approximately 80% ammonium bisulfate. The current density at anode ranges from 80 to 120 amp/dm². The cathode current density being 8 to 10 amp/dm². The cathodes are wrapped by blue asbestos rope as diaphragm. Working temperature range is between 25 to 30°C and the addition agent being 0.1 to 0.5 g/l.

Example

An aqueous solution of ammonium sulfate and sulfuric acid of the composition to give 80% W/V ammonium bisulfate with addition of 0.3 g/l of urea is electrolysed in an electrolyser made of a non conducting material such as ceramic, plastic, hard rubber, glass between platinum and lead electrodes as anode and cathode respectively. The cathode is covered by a rope of blue asbestos. The anode current density is 100 amp/dm² and cathode current density is 8 amp/dm². The temperature is maintained at 30°C by circulation of chilled water through glass cooling coils immersed in the cell. When the slurry makes its appearance in the cell the electrolyte from storage tank is fed continuously and the slurry is tapped out from the bottom of the electrolyser in such a way that the composition in the electrolyser is maintained. The composition of the solution in the electrolyser is maintained at ammonium persulfate 8%, ammonium sulfate 29% and sulfuric acid 21.5% during electrolysis. The effluent slurry is centrifuged and washed with water at 0°C to 5°C to free the solid from absorbed acid. Finally the product is dried in vacuum drier. The purity of the product is 98%, the balance being bound water.

In bench scale experiment the cell was of glass container of capacity 1 litre containing 600 cc electrolyte of 80% ammonium bisulfate with 0.3 g/l urea. Current was 12 amps at anode current density 100 amp/dm² and cathode current density 8 amp/dm². Temperature was 30°C. The current efficiency obtained was 80%.

Following are among the main advantages of the invention:

The product obtained is pure straight from the cell in one step, which reduces the number of unit operations to a minimum without impairing the current yield or the quality of the product.

Dated this 25th day of March, 1975.

Sd/-
Asstt. Patents Officer,
Concil of Scientific & Industrial Research

THE PATENTS ACT 1970

COMPLETE SPECIFICATION

SECTION 10

"A PROCESS FOR THE PREPARATION OF SOLID AMMONIUM PERSULPHATE"

**COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, Rafi Marg,
New Delhi, India - an Indian Registered body incorporated under
the registration of Societies Act (Act XXI of 1860).**

**The following specification describes and ascertains the
nature of this invention and the manner in which it is to be
performed :-**

**This is an invention by Mahadev Govind Potdar, Scientist;
Rangachari Srinivasan, Scientist and Handady Venkatakrisna Udupa,
Director, all of Central Electrochemical Research Institute,
Karaikudi-623 006, Tamil Nadu, all Indian citizens.**

This invention relates to a process for the preparation of solid ammonium persulphate.

Hitherto solid ammonium persulphate has been prepared by indirect method only i.e., an aqueous solution of ammonium sulphate and sulphuric acid is electrolysed between platinum anode and lead cathode. The electrolysis is carried out in a suitable vessel called electrolyser which is either divided type or undivided type. The concentration of the aqueous solution used is very dilute and on electrolysis a concentrating ^{led} solution of ammonium persulphate is obtained. This solution is taken out of the electrolyser as effluent and then further treated to get solid ammonium persulphate. The treatment lies in further concentration by evaporation or by chilling the solution to low temperature to get solid ammonium persulphate as first ^{fraction} fraction.

This is open to the objection that the unit operation of heating for concentration of the solution tends to decompose the heat sensitive ammonium persulphate in one process and in the other process refrigeration involves an additional ⁻⁹¹ cooling step. Also the refrigerated product contains 5 to 10% $(\text{NH}_4)_2\text{SO}_4$ as impurity.

The object of this invention is to obviate the above mentioned difficulties by developing a process for making solid ammonium persulphate directly from an electrolytic cell and with a higher purity than is obtainable by chilling the electrolyte in the known process.

The main finding of the invention is that an aqueous solution of ammonium sulphate and sulphuric acid taken in equimolar proportions in order to give approximately 80% ammonium bisulfate, can be electrolysed between platinum and lead electrodes in an undivided electrolytic cell to give solid ammonium persulphate directly from the electrolytic cell in the form of a slurry.

When ammonium bisulfate solutions of approximately 80% w/v concentration are electrolysed between platinum anode and lead cathode the anode current density being approximately 100 amp/dm² and the

cathode current density being approximately 10 amp/dm², at a temperature of about 30°C, addition agent in the form of urea being used at a concentration of approximately 0.3 g/l, solid ammonium persulphate is obtained directly from the electrolytic cell in the form of a slurry.

The composition of electrolyte i.e., ammonium sulphate and sulphuric acid can be such as to give approximately 80% ammonium bisulfate. The current density at anode ranges from 80 to 120 amp/dm², the cathode current density being 8 to 10 amp/dm². The cathodes are wrapped with blue asbestos rope as diaphragm. Working temperature range is between 25 to 30°C and the addition agent being 0.1 to 0.5 g/l.

According to the present invention, there is provided a process for preparation of solid ammonium persulphate by the electrolysis of an aqueous solution of ammonium sulfate and sulphuric acid between platinum anode and lead cathode characterised in that these ingredients are mixed in equimolar concentration to give a solution of ammonium bisulfate in the range of 70% to 90% wt/Vol. and electrolysed in the presence of an addition agent in the form of urea. 80% w/v concentration is preferred along with an addition agent in the form of urea in 0.1 to 0.5 g/l concentration but preferably in 0.3 g/l concentration and the cathode current density being 8 to 10 amp/dm² but preferably 10 amp/dm². The temperature is maintained within 25 to 35°C and the conditions are such that solid ammonium persulphate starts forming in the cell during passage of current. The electrolysis is carried out in an undivided type of electrolysing cell. The solid is tapped continuously or by batchwise collection for further processing like washing, drying, etc.

The novelty of this process is that solid ammonium persulphate is obtained continuously directly from the cell unlike other processes in vogue wherein crystallisation is an additional unit operation subsequent to electrolysis.

Example

An aqueous solution of ammonium sulphate and sulphuric acid of the composition to give 80% w/v ammonium bisulfate with addition of 0.3 g/l of urea is electrolysed in an electrolyser made of a non-

conducting material such as ceramic, plastic, hard rubber, glass between platinum and lead electrodes as anode and cathode respectively. The cathode is covered by a rope of blue asbestos. The anode current density is 100 amp/dm^2 and cathode current density is 8 amp/dm^2 . The temperature is maintained at 30°C by circulation of chilled water through glass cooling coils immersed in the cell. When the slurry makes its appearance in the cell the electrolyte from storage tank is fed continuously and the slurry is tapped out from the bottom of the electrolyser in such a way that the composition in the electrolyser is maintained. The composition of the solution in the electrolyser is maintained at ammonium persulphate 8%, ammonium sulfate 29% and sulfuric acid 21.5% during electrolysis. The effluent slurry is centrifuged and washed with water at 0°C to 5°C to free the solid from absorbed acid. Finally the product is dried in vacuum drier. The purity of the product is 98% the balance being bound water.

In bench scale experiment the cell was of glass container of capacity 1 litre containing 600 cc electrolyte of 80% w/v ammonium bisulphate with 0.3% g/l urea. Current was 12 amps at anode current density 100 amp/dm^2 and cathode current density 8 amp/dm^2 . Temperature was 30°C . The current efficiency obtained was 80%

Following are among the main advantages of the invention:

The product obtained is pure straight from the cell in one step, which reduces the number of unit operations to a minimum without impairing the current efficiency or the quality of the product.

A process has been developed for the electrochemical preparation of solid ammonium persulphate directly from the electrolytic cell. This has the advantage of reducing the unit operations of concentration and crystallisation. The crystal size also can be controlled in the operation of the electrolysis to facilitate filtration and drying. The purity of the product obtained is 98% and thus this eliminates additional recrystallisation step also. It is also possible to work out this process either as a continuous one or batchwise without impairing any of the parameters.

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We claim:

- 1) A process for the preparation of solid ammonium persulphate by the electrolysis of an aqueous solution of ammonium sulphate and sulphuric acid between platinum anode and lead cathode characterised in that these ingredients are mixed in equimolar concentration to give a solution of ammonium bisulfate in the range of 70 to 90% W/V and electrolysed in the presence of an addition agent in the form of urea.
- 2) A process as in claim 1 wherein the current density is 80 amp/dm² to 120 amp/dm² at the anode and 8-10 amp/dm² at cathode.
- 3) A process as in the claims 1 and 2 wherein blue asbestos rope is wrapped over lead cathode to serve as a diaphragm.
- 4) A process as in claims 1 to 3 wherein the electrolysis is carried out at a temperature range between 25°C to 35°C.
- 5) A process as in claims 1 to 4 wherein the solid ammonium persulphate formed as slurry in the cell is removed continuously or at intervals in batches.
- 6) A process as in claims 1 to 5 wherein the electrolysis is carried out in an undivided type of electrolysing cell.
- 7) A process as in claims 1 to 6 wherein urea is added to the electrolyte in the range 0.1 gm/l to 0.5 g/l as addition agent.
- 8) A process for the preparation of solid ammonium persulphate substantially as hereinbefore described.

Dated this 10th day of June 1976.


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