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A PROCESS FOR THE ELECTROLYTIC PREPARATION OF CUPROUS OXIDE.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, OLD MILL ROAD, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860).

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

This is an invention by RAMANATHAN VISWANATHAN, SRINIVASA SAMPATH, HANDADY VENKATAKRISHNA UDUPA, AKKUNDI JOGARAO and BIMAN BIHARI DEY, all of the Central Electro Chemical Research Institute, Alagappa College P. O., Karaikudi, S. Rly., Madras, India, all Indians.

This invention relates to a process for the electrolytic preparation of cuprous oxide.

Cuprous oxide was prepared for the first time by the electrolytic method by Lorenz in 1896. Other workers have tackled the problem from a purely academic view-point. Important process details so essential for manufacture on a practical scale have not been worked out. There are references in literature to the preparation of the yellow variety of cuprous oxide by continuous addition of a protein-containing colloid during electrolysis. A recent process claims an energy consumption of 1.25 kwhr./kg.

The present invention consists in feeding pre-heated electrolyte containing not less than 10 per cent sodium chloride by weight and 0.3 to 1 gram per litre alkali (expressed as sodium hydroxide) into the cathode chambers of an electrolytic cell in which sheet copper is used as cathode and pure or refined copper sheet is employed as anode and wherein the rate of flow of the electrolyte is such that the alkalinity of the bath is always maintained at the specified optimum range, *viz.*, 0.3 to 1 gpl. expressed as NaOH.

In the following paragraphs the details of the process worked out and of the continuous unit employed are set out clearly with the help of the accompanying drawings in which Figure 1 is a flow diagram of the process, showing the location of the electrolytic cell and its accessory equipment, Figure 2 shows the circulation arrangement to maintain the flow of electrolyte into the cathode compartments, Figures 3 and 4 are sectional views of the electrolytic cell.

The Electrolytic Cell.—The cell (A) is made of reinforced cement concrete and is a square container (Z) (could be rectangular) provided with a conical bottom (P). An opening (Q) at the bottom serves for the removal of the product as it is being formed. A lid (B) made of teakwood covers the cell and carries suitable slots (R) for the introduction of the electrodes (C & D) and also a supporting framework for the diaphragms (E). Glass or glass-lined equipment, asphalt-coated wooden tanks, ceramic ware or metal containers with suitable protective coating could also be used as cells.

Auxiliary equipment.—(i) *Heating tank*—Cold electrolyte was fed into this 30-litre tank (F) of porcelain or of any other suitable material where three immersion heaters (G) (2 kw. capacity each) served to raise the temperature to the desired level.

(ii) *Pump (H)*—This pump made of naval bronze driven by a ½ H.P. motor was used to pump the hot solution from the heating tank to the constant head tank (I).

(iii) *Constant head tank (I)*—This was a tall cylindrical tank 2" in diameter and lagged suitably with asbestos to minimise radiation losses.

(iv) *Filter press (J)* 6" × 6" plate and frame press for filtering the product from the cell.

Operation.—The constant head tank (I) feeds the hot electrolyte equally into the five cathode compartments (W) at a steady predetermined rate. The rate of feeding of electrolyte is a function of the ratio of anolyte to catholyte and the current strength. Electrolyte containing cuprous oxide in suspension is withdrawn from the bottom (P) of the cell in order to maintain a constant level in the cell. The product may be settled, before filtration using a filterpress or a centrifuge. The clear liquor from the settler and the filtrate are fed back into the cell (A) after heating to the required temperature. The product in the filter (J) is washed free of adhering electrolyte and dried (drier, K) at about 100°C preferably under reduced pressure. The dry solid is pulverized and screened (L) before packing (M).

Conditions of Electrolysis.—Electrolyte contains 10 per cent sodium chloride by weight and one gram per litre of sodium hydroxide. Electrolysis is carried out at a temperature of 80°C, the solution being heated either by electricity or by steam.

Electrode Materials.—Sheet copper is used as cathode (C). Either pure or refined copper sheet is employed as anode (D). The square electrode sheets (9" × 9") each are suspended from a copper flat (S) fixed to the busbar (T) at one end (U) and resting on the cell lid (B) at the other.

Current Density.—Anodic current density is maintained at 5 amp. per sq. dm. As there is one cathode more than the number of anodes used the cathode current density is less. In this particular cell, it is 4 amp./dm². The current concentration is about 7 to 8 amps./litre.

Ratio of anolyte to catholyte.—This varies with the design of the cell and is one of the factors controlling the rate of circulation of the electrolyte. In this cell the volume of catholyte used was 4 litres and that of anolyte was 22 litres.

Rate of Circulation.—There is a build-up of alkali at the cathode during electrolysis and thus has to be dissipated into the main body of the anolyte which gets depleted in alkali. The rate of flow of the electrolyte is so adjusted that the alkalinity of the bath is always maintained at 1 gpl. NaOH. In the cell (A) described a rate of 30 litres per hour was found to be optimum.

Diaphragm material.—Cloth diaphragm (E) was found to be most suitable for the purpose. It effectively prevents cuprous oxide from moving to the cathode and getting reduced as spongy copper but allows the electrolyte to flow through freely. Also it does not offer too great a resistance for the passage of current. Drill cloth stitched on to wooden frames (V) fitted to the lid (B) (each cathode compartment (W) was 9" × 9" × ½") was found to be ideal for the purpose.

Circulation arrangement.—(N, Figure II) From the constant head tank (I) the specified rate of flow was maintained into the cell (A) by suitably adjusting the level of the liquid in the head tank. The feed of the electrolyte into the cathode compartments (W) was effected by means of five tubes (Y) branching at regular intervals from a common header tube (Z) connected to the head tank (I).

EXAMPLE.

The following are the results in a typical experiment :

Current passed	180 amp.	
Voltage of the bath	1.75 volts	
Duration of electrolysis	5 hours	
Temperature	80°C	
Anodic current density	5 amp./dm ² .	30
Weight of product	5 lbs.	
Purity of the product	96 per cent.	
Current efficiency	94.5 per cent.	
Colour of the dry sample	bright red	35
Energy consumption	0.325 Kwhr./lb.	

The sample meets the requirements of Indian Standard Specifications and is fit for use in antifouling compositions or for any other purpose. The current efficiency of the process is over 94 per cent and the energy consumption will not exceed 0.35 kwhr./lb. 40

We claim :

1. A process for the electrolytic preparation of cuprous oxide which consists in feeding pre-heated electrolyte containing not less than 10 per cent sodium chloride by weight and 0.3 to 1 gram per litre alkali (expressed as sodium hydroxide) into the cathode chambers of an electrolytic cell in which sheet copper is used as cathode and pure or refined copper sheet is employed as anode and wherein the rate of flow of the electrolyte is such that the alkalinity of the bath is always maintained at the specified optimum range *viz.*, 0.3 to 1 gpl. expressed as NaOH. 45

2. A process as claimed in Claim 1 wherein constant feeding of electrolyte into the cathode chambers is achieved by means of tubes branching from a common header tube connected to a constant head tank, the level of the liquid in which maintains the desired rate of flow into the cell. 50

3. A process as claimed in claim 1 wherein the electrolysis is carried out in an electrolytic cell with a conical bottom permitting the removal of the product continuously as it is formed. 55

4. A process as claimed in the preceding claims wherein the electrolysis is carried out at a temperature of 80°C. 60

5. A process as claimed in any of the preceding claims wherein the rate of feeding of electrolyte is a function of the ratio of anolyte to catholyte and the current strength. 65

6. A process as claimed in any of the preceding claims wherein the electrolyte (anolyte) along with the product is tapped at the same rate as it is fed, to maintain the required alkalinity in the anolyte.

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Price : TWO RUPEES.