GOVERNMENT OF INDIA : THE PATENT OFFICE, 214, LOWER CIRCULAR ROAD, CALCUTTA-17.

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IMPROVEMENTS IN OR RELATING TO THE PRODUCTION OF COPPER POWDER FROM BYPRODUCT COPPER OXIDES OBTAINED FROM THE COPPER WIRE AND TUBE DRAWING AND SHEET ROLLING INDUSTRY.

PROVISIONAL SPECIFICATION

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

This is an invention by HANDADY VENKATAKRISHNA UDUPA, Scientist, and PENNAGARAM VYASA RAO VASUDEVA RAO, Scientist, both of the Central Eelectrochemical Research Institute, Karaikudi 3, Madras State, India, both Indian citizens.

The following specification describes the nature of this invention

This invention relates to improvements in or relating to production of copper powder from byproduct copper compounds such as the oxides obtained from copper sheet rolling and copper wire and tube drawing industries

Hitherto it has been the practice to convert the oxides, hydroxides etc, into copper sulphate or similar copper salts by dissolving the byproduct oxides in suitable acids

This is open to the objection that large volume of solutions will have to be handled and acid will have to be used Processing steps for making copper powder are hereby increased also

Reducing the oxides directly into powder by simple electrochemical reduction eliminates the intermediate steps of bringing the copper compound into solution and electrolysing subsequently and gives good grade of copper powder suitable for most of the applications

The object of this invention is (1) to utilize as a solid depolarizer, large amounts of copper oxide which become available on an industrial scale to produce copper powder directly from the same by eliminating the intermediate step of preparing the copper salt solution and in particular (2) to produce copper powder from byproduct copper oxides obtained from copper sheet metal rolling, wire and tube drawing industries

To these ends, the invention broadly consists in a method of direct electrochemical reduction of copper oxides and similar compounds of copper to copper powder in a suitable cell arrangement using alkaline electrolyte

The cell arrangement consists of a copper, mild steel, stainless steel, nickel or nickel-plated container in which the copper oxide and/or similar compound of copper is contained in a powder form of suitable mesh size The container itself is used as the cathode The anode assembly consists of a mild steel, nickel-plated mild steel, nickel or stainless steel mesh suitably disposed over the bed of the powder and mounted on a frame in such a way that, while immersed in an electrolyte of sodium hydroxide or potassium hydroxide over the bed of the copper oxide and/or similar compound of copper, it will facilitate uniform distribution of current on the cathode surface

The following typical examples illustrate the invention and the manner of carrying out the same is also given

Wt of cupric oxide taken (g)	Concentration of alkali (%)	Cell voltage (volts)	Amp-hr passed	Cathode efficiency (%)	Sample passing 150 mesh (%)
2 5	5	2 1	1 75	100	100
450	20	1 8	300	98	98
500	10	1 9	350	95	90

In an experiment, a circular mild steel container of 12' diameter was used as cathode, nickel plated mild steel was used as anode, which was mounted on an insulating frame and disposed 1" over the bed of copper oxide of —150 mesh (the thickness of the bed being about 1 cm) in an electrolyte of 20% sodium hydroxide The current density employed was 10 amp/dm² calculated on the basis of apparent surface area 10% excess of the total theoretical current required to reduce the cupric oxide taken was passed The cell voltage was 1 5-2V. At the end of the experiment, the copper powder was discharged from the cell, washed free from electrolyte, stabilised and dried The conversion efficiency was almost 100% and current efficiency ranged from 85-90%. In order to improve the quality of the copper powder so obtained, the same was given an annealing treatment in a hydrogen atmosphere and then stabilised and dried

The following are among the main advantages of the invention :

1 The byeproduct copper oxide obtained is directly converted into copper powder The usual step of obtaining a soluble salt of copper and then electrolysing is avoided

2 The particle size of the copper powder obtained is dependent on the particle size of the copper oxide used in the reduction

3 The alkali can be used continuously by making up only the loss during processing

4 Large quantities of copper powder could be produced with minimum of control of process variables and in required particle size by choosing appropriate fractions of copper oxide

R BHASKAR PAI

Patents Officer,

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH.

Dated this 7th day of May 1965.

COMPLETE SPECIFICATION.

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

This is an invention by HANDADY VENKATAKRISHNA UDUPA, Scientist and PENNAGARAM VYASA RAO VASUDEVA RAO, Scientist, both of the Central Electrochemical Research Institute, Karaikudi, S Rly, Madras State, India, both Indian citizens

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

This invention relates to improvements in or relating to production of copper powder from byproduct copper compounds such as oxides obtained from copper wire and tube drawing and sheet rolling industries and from other industries,

Price : TWO RUPEES.

Hitherto, it has been the practice to convert the oxides, hydroxides etc., into copper sulphate or similar copper salts by dissolving the byproduct oxides in suitable acids and electrolysing the resulting solution.

The main disadvantage in the older processes is that large volume of solutions will have to be handled and acid will have to be used. Processing steps for making copper powder are thereby increased also.

The object of this invention is (1) to utilise as a solid depolarizer, large amounts of copper oxide which become available in an industrial scale to produce copper powder directly from the same and (2) to produce copper powder from byproduct copper oxide obtained from copper sheet metal rolling, wire and tube drawing industries and from other sources.

According to the present invention, the process for the electrochemical production of copper powder from byproduct copper oxides obtained from the copper wire and tube drawing and sheet rolling industry or the chemical industry comprises the electrochemical reduction of the solid copper oxide, kept as a sediment on the cathode substrate of an electrolytic cell containing an aqueous alkaline electrolyte (preferably NaOH solution) keeping an alkali-resistant anode immersed in the electrolyte but above the sediment.

The particles size of the copper powder obtained is controlled by choosing appropriate size fraction of the copper oxide as illustrated herein below :

Particle size fraction and Weight of the oxide taken for reduction.		Particle size fraction and weight of copper powder obtained.
−150 to + 200	100 g.	−150 to + 200/−70 g.
−200 to + 300	100 g.	−200 to + 300/−72 g.
−300	100 g.	−300 /−72 g.

The alkaline electrolyte may be used continuously by making up the loss during processing.

The copper powder is annealed in an atmosphere of hydrogen and then stabilised in the usual way.

Fine powder of the byproduct copper oxide is kept as a sediment at the bottom of the cell and the cell is made the cathode. Sodium hydroxide solution serves as the electrolyte and as the current is passed, the oxide gets reduced to copper powder.

This process of direct reduction offers certain advantages, *viz.*, (a) this eliminates the intermediate steps of bringing the copper compound into solution and electrolysing subsequently. (b) This avoids handling of large volume of acids and (c) the process is very easy to operate and good grade of copper powder suitable for most of the aplications, can be produced by this process. The particle size of the copper powder obtained is dependent on the particle size of the oxide used in the reduction.

Good grade copper powder can be produced by this process.

The conversion efficiency is almost 100% with a current efficiency ranging from 85 to 90%.

In this process, copper powder is produced by direct electrochemical reduction of copper oxide and similar compounds of copper in a suitable cell arrangement using an alkaline electrolyte. The cell arangement consists of a copper, mild steel, stainless steel, nickel or nickel plated mild steel container in which the copper oxide and/or similar compound of copper is contained in a powder form of suitable mesh size. The container itself is used as the cathode. The anode assembly consists of a mild steel, nickel-plated mild steel, nickel or stainless steel mesh suitably disposed over the bed of the powder and mounted on a frame in such a way that, while immersed in an electrolyte of sodium or potassium hydroxide over the bed of the copper oxide and/or similar compound of copper, it will facilitate uniform distribution of current on the cathode surface. A circular mild steel container of 12" diameter was used as cathode in an experiment and nickel-plated mild steel was used as anode which was mounted on an insulating frame and disposed 1" over the bed of copper oxide of −150 mesh in an electrolyte of 20% sodium hydroxide. The current density employed was 10 amp/dm² and 10% excess of the total theoretical current required to reduce the cupric oxide taken was passed. At the end, the copper powder was discharged from the cell, washed free from electrolyte, stabilised and dried. In order to improve the quality of the copper powder so obtained, the same was given an annealing treatment in hydrogen atmosphere and then stabilised and dried.

The following typical examples are given to illustrate the invention :

EXAMPLE I

Weight of cupric oxide taken (g)	Concentration of alkali (%)	Cell voltage (volts)	Amp-hr. passed	Cathode efficiency (%)	Sample passing 150 mesh (%)
2.5	5	2.1	1.75	100	100
450	20	1.8	300	98	98
500	10	1.9	350	95	90

EXAMPLE II

Weight of the oxide taken	2.5 kg
Thickness of the bed	1 cm
Electrolyte	20% sodium hydroxide
Current density	10 amp/dm²
Cell voltage	1.5 to 2.0 V
Conversion efficiency	100%
Current efficiency	85–90%

The main advantages of this process are : (a) The by product copper oxide obtained is directly converted into copper powder. The usual step of obtaining a soluble salt of copper and then electrolysing is avoided. (b) The particle size of the copper powder obtained is dependent on the particle size of the copper oxide used in the reduction. (c) The alkali can be used continuously by making up only the loss during processing. (d) Large quantities of copper powder could be produced with minimum control of process variables and in required particle size by choosing appropriate fractions of copper oxide or scales.

This invention enables the production of copper powder by direct reduction of copper oxides obtained from the copper wire and tube drawing and sheet rolling industry. Reducing the oxides directly by simple electrochemical reduction in alkaline medium eliminates the intermediate steps of dissolving the copper compound followed by electrolysis of the resulting solution. Copper, mild steel, nickel or nickel-plated mild steel container was used as the cell and the copper compound was kept as a sediment at the bottom of the cell. The cell was made as the cathode and a mild steel, nickel-plated mild steel, nickel or stainless steel mesh, mounted on an insulating frame, suitably disposed over the bed of the powder so as to facilitate uniform current distribution served as the anode. At the end, the copper powder was taken out from the cell, washed free from electrolyte,

stabilised and dried. The conversion efficiency was almost 100% and current efficiency ranged from 85-90%. In order to improve the quality of the copper powder so obtained, the same was given an annealing treatment in hydrogen atmosphere.

We claim :

1. A process for the electrochemical production of copper powder from byproduct copper oxides obtained from the copper wire and tube drawing and sheet rolling industry or the chemical industry which comprises the electrochemical reduction of the solid copper oxide, kept as a sediment on the cathode substrate of an electrolytic cell containing an aqueous alkaline electrolyte (preferably NaOH solution) keeping an alkali-resistant anode immersed in the electrolyte but aobve the sediment.

2. A process as claimed in Claim (1) wherein the particle size of the copper powder obtained is controlled by choosing appropriate size fraction of the copper oxide as illustrated herein below :

Particle size fraction and Weight of the oxide taken for reduction.		Particle size fraction and weight of copper powder obtained.
—150 to + 200	100 g.	—150 to + 200 ~70 g.
—200 to + 300	100 g.	—200 to + 300 ~72 g.
—300	100 g.	—300 ~72 g.

3. A process as claimed in Claim 1 or 2 wherein the alkaline electrolyte is used continuously by making up the loss during processing.

4. A process as claimed in any of the preceding claims wherein the copper powder is annealed in an atmosphere of hydrogen and then stabilised in the usual way.

5. A process for making copper powder from byproduct copper oxides substantialy as hedeinbefore described.

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Dated this 19th day of February 1966.

MGIP Sant.—30 Patent/67—31-3-69—100.