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

Specification No. 102677. Application No. 102677 dated 25th November 1965. Complete Specification left on 24th August 1966. (Application accepted 23rd August 1967.)

Index at acceptance—70C 4+5 [LVIII(5)],

PROVISIONAL SPECIFICATION.

IMPROVEMENTS IN OR RELATING TO THE PRODUCTION OF SILVER POWDER SUITABLE FOR MAKING CONDUCTING INKS

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),

The following specification describes the nature of this Invention.

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

This invention relates to improvement in or relating to production of silver powder suitable for making conducting inks.

Hitherto the practice has been to suspend silver powder in a suitable medium which is volatile and evaporates on exposure to air and leaves the suspended silver powder behind. A suitable binder which is soluble in the medium keeps the silver powder particles bound together so that when they are dry, they make a continuous contact. Finding a suitable medium and also preparation of very fine silver powder that can be kept suspended in the medium have been major difficulties encountered. The resistance of the dried silver film is also considerably high.

By the present method, it has been found possible to prepare very fine silver powder and by suspending the same in a suitable medium (medium+binder hereafter called vehicle) it has also been possible to prepare a conducting ink which possesses superior characteristics to those available at present.

The object of the invention is (1) to prepare very fine silver powder suitable for suspending in (2) a proper vehicle for preparing conducting inks.

To these ends, the invention broadly consists in (a) method for the preparation of very fine silver powder by direct reduction of silver oxide or any other suitable compound of silver, (b) design of proper cell arrangement for achieving (a) (c) a method for preparing conducting inks from the silver powder obtained from (a); and (d) suitable vehicles for preparing conducting inks as at (c).

The cell arrangement consists of a silver, stainless steel or silver plated copper container in which the silver oxide or any other suitable compound of silver is kept as a layer at the bottom. The container itself is used as the cathode. The anode assembly consists of a nickel, stainless steel or platinum wire mesh or sheet perforated suitably, disposed over the bed of the silver compound and mounted on a frame in such a way that while kept in an electrolyte of potassium or sodium hydroxide solution of suitable strength will allow uniform current distribution on the cathode surface. The anode is kept in such a way that the cell voltage is as low as possible. When current is passed, the silver compound in contact with the bottom of the container gets converted to silver powder. The powder is discharged from the cell, washed free from the electrolyte and dried in an air oven below 100°C.

The method of making the conducting ink is as follows:

The silver powder is sieved through a 325 mesh sieve to remove any coarse particles than—325 size. A suitable vehicle (medium+binder) is prepared and the silver powder is added to the same in a predetermined ratio and mixed thoroughly by a suitable mixing mechanism. The vehicle is prepared as follows:

An organic solvent like benzene, trichloroethylene, hexachloromethylene, cyclohexonone or any other suitable solvent is taken and a plastic such as methyl methacrylate, polystyrene, polyvinyl chloride or a polyamide of high molecular weight is added to the same to prepare a solution which when exposed to atmosphere or heated evaporates quickly leaving behind a uniform continuous layer of the plastic material.

The following typical example is given to illustrate the invention:

EXAMPLE

In an experiment to prepare silver powder, the following conditions were adopted:

1. Material:	Silver oxide—50 g.
2. Base:	Silver plated copper
3. Electrolyte:	10 per cent sodium hydroxide
4. Current passed.	5 amp.
5. Current efficiency:	98 per cent.
6. Conversion efficiency:	100 per cent.
7. Particle size:	—325 100 per cent.

Typical formula of the vehicle:

Perspex powder.	2 g.
Trichloroethylene:	100 cc.

Typical formula for the ink:

Vehicle as prepared above:	50 cc.
Silver powder as prepared above: made into a mixture	25 g.

Application:

The mixture as above is well stirred before applying. The powder as prepared above can also be used for preparing silver impregnated carbon brushes, etc.

The following are among the main advantages of the invention.

(a) This method of making fine silver powder is itself novel and does not appear to have been adopted earlier.

(b) Very fine silver powder suitable for making conducting inks and products such as carbon brushes etc., can be produced.

(c) Conducting ink possessing superior qualities can be prepared with the silver powder obtained as above.

(d) A suitable vehicle has been worked out for keeping the silver powder in suspension.

(e) The contacts made with this ink have lower resistance than those made with the conducting ink prepared from silver powder produced by conventional processes.

R. BHASKAR PAI,

Patents Officer,

Council of Scientific and Industrial Research.

Dated this 10th day of November 1965.

Price : TWO RUPEES.

COMPLETE SPECIFICATION.

IMPROVEMENTS IN OR RELATING TO THE PRODUCTION OF SILVER POWDER SUITABLE FOR MAKING CONDUCTING INKS.

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).

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 HANDADY VENKATAKRISHNA UDUPA, Scientist, and PENNAGARAM VYASA RAO VASUDEVA RAO, Scientist, both of the Central Electrochemical Research Institute, Karaikudi-3, India; both Indian citizens.

This invention relates to improvements in or relating to the production of silver powder suitable for making conducting inks and products such as silver paint, silver incorporated carbon brushes and sintered compacts suitable for use in alkaline batteries and fuel cells.

Hitherto the practice has been to prepare silver powder either by chemical displacement or by electrodeposition from silver salt solution.

The main drawback of the hitherto known processes has been that the particle size and shape of silver powder prepared by these processes have not been found to be suitable for the above stated purposes.

The object of this invention is to prepare very fine silver powder mainly suitable for making conducting ink and production of products such as sintered silver compacts for alkaline batteries and fuel cells and silver incorporated carbon brushes.

We have found that fine silver powder can be prepared by direct reduction of silver compounds such as silver oxide by keeping the same as a sediment on suitable metal (silver, stainless steel, nickel or silver coated copper) cathode in an alkaline electrolyte.

According to the present invention, the process for preparing silver powder suitable for making products such as conducting ink, silver paint, silver incorporated carbon brushes and sintered compacts suitable for use in alkaline batteries and fuel cells consists in the direct electro-chemical reduction of solid silver compounds such as the oxide wherein the silver compounds are kept as a sediment at the bottom of a silver, stainless steel, nickel or silver coated copper container which serves as the cathode, in an alkaline electrolyte of strength up to 40 per cent. sodium hydroxide employing current density up to 50 amp/dm^2 but preferably 10 amp/dm^2 and wherein nickel or stainless steel or platinum wire mesh or perforated sheet kept immersed in the electrolyte, maintaining uniform current distribution on the entire cathode surface, is used as the anode.

The alkaline electrolyte is repeatedly used, only electrolyte lost during the process is made up by adding fresh electrolyte. Very fine silver powder suitable for the above purposes can be recovered after the reduction is over.

The conversion efficiency is 100 per cent. with a current efficiency of 98-100 per cent.

Very fine silver powder can be prepared by this method without losing much as a coarse fraction unsuitable for purpose stated above.

This method of making silver powder is itself novel and does not appear to have been adopted earlier.

The silver powder prepared by this method can be used for making conducting ink of superior quality as well as for preparing sintered compacts for alkaline batteries and fuel cells and silver incorporated carbon brushes.

Thus, for preparing silver powder of a very fine particle size by reducing silver compounds, such as oxide, cathodically in an alkaline electrolyte, the cell arrangement consists of a silver, stainless steel, nickel or silver plated copper container in which the silver oxide or any other suitable compound of silver is kept as a layer at the bottom. The container itself is used as the cathode. The anode assembly consists of a nickel, stainless steel or platinum wire mesh or perforated sheet, suitably disposed over the bed of the silver compound and mounted in such a way that while kept in an electrolyte of potassium or sodium hydroxide solution of suitable strength, will facilitate uniform current distribution on the cathode surface. The anode is kept in such a way that the cell voltage is as low as possible. When current is passed, the silver compound in contact with the bottom of the container gets converted to silver powder. The powder is then taken out from the cell, washed free from alkali and dried in an air oven below 100° C.

The following are the typical examples:

A. PREPARATION OF SILVER POWDER:

Example I.

Material taken:	2.5 g. silver oxide.
Base:	Nickel.
Electrolyte:	4 per cent. NaOH.
Current density:	50 amp/dm^2.
Current passed:	8 amps.
Cell voltage:	4.1 volts.
Current efficiency:	94.8 per cent.
Weight of silver powder obtained:	2.11 g.
Apparent density:	0.9078.

Example II.

Material taken:	2.5 g. silver oxide.
Base:	Nickel.
Electrolyte:	20 per cent. NaOH.
Current density	25 amp/dm^2.
Current passed:	3.8 amps.
Cell voltage:	3.8 volts.
Current efficiency:	99.3 per cent.
Weight of silver powder obtained:	2.321 g.
Apparent density:	0.2737 g/cc.

Example III.

Material taken:	2.5 g. silver oxide.
Base:	Nickel.
Electrolyte:	40 per cent. NaOH.
Current density:	2 amp/dm^2.
Current passed:	0.3 amps.
Cell voltage:	2.4 volts.
Current efficiency:	100 per cent.
Weight of silver powder obtained:	2.311 g.
Apparent density:	1.202 g/cc.

Example IV.

Material taken:	50 g. silver oxide.
Base:	Silver plated copper.
Electrolyte:	10 per cent. NaOH.
Current passed:	5 amps.
Current efficiency:	98 per cent.
Conversion efficiency:	100 per cent.
Particle size-325:	100 per cent.

The following are among the main advantages of the invention:

(a) This method of making fine silver powder is itself novel and does not appear to have been adopted earlier.

(b) Very fine silver powder suitable for making conducting inks and production of products such as carbon brushes, sintered compacts for alkaline batteries and fuel cells, etc., can be produced by this process.

(c) Conducting ink possessing superior qualities can be prepared by using the silver powder as obtained above.

(d) This process of preparation of very fine silver powder is simple.

(e) Silver powder of uniform particle size is formed without much of a fraction of coarse particles.

The invention enables to the preparation of very fine silver powder suitable mainly for making conducting ink. In this method, fine silver powder is prepared by direct reduction of silver compounds such as the oxide. This process of preparation of silver powder is very simple and is itself novel and does not appear to have been adopted earlier. The powder obtained by this process is suitable for making conducting ink and production of products such as carbon brushes and sintered compacts suitable for alkaline batteries and fuel cells etc. In addition, the ink prepared by using the powder obtained by this process possesses superior qualities and provides good contacts with a very low resistance. The silver compound is kept as a sediment on the metal cathode in an alkaline electrolyte and directly reduced to the metal powder, the particle size of which can be controlled by controlling the particle size of the starting material. The conversion efficiency is 100 per cent. with a current efficiency of 98-100 per cent. The powder after the reduction is over, is taken out of the cell, washed free from alkali and dried in an air oven below 100° C. Conducting ink having superior quality is prepared using the silver powder obtained by the above process by suspending the same in a suitable medium or vehicle. The main advantage of this process lies in the simplicity of the process and the fineness of the powder obtained which on suspending in a suitable medium gives conducting ink having superior properties, characterised by very low resistance.

We claim:

1. A process for making silver powder suitable for making products such as conducting ink, silver paint, silver incorporated carbon brushes and sintered compacts suitable for use in alkaline batteries and fuel cells which consists in the direct electrochemical reduction of solid silver compounds such as the oxide wherein the silver compounds are kept as a sediment at the bottom of a silver, stainless steel, nickel or silver plated copper container which serves as the cathode, in an alkaline electrolyte of strength up to 40 per cent. sodium hydroxide employing current density up to 50 amp/ dm^2 but preferably 10 amp/dm^2 and wherein nickel or stainless steel or platinum wire mesh or perforated sheet kept immersed in the electrolyte, maintaining uniform current distribution on the entire cathode surface, is used as the anode.

2. A process as claimed in Claim 1, wherein the alkaline electrolyte is repeatedly used, and the electrolyte lost during the process is made up by adding fresh electrolyte.

3. A process for making silver powder from silver compounds substantially as hereinbefore described.

4. Silver powder suitable for making products such as conducting ink, silver paint, silver incorporated carbon brushes and sintered compacts suitable for use in alkaline batteries and fuel cells, whenever produced according to a process substantially as hereinbefore described.

R. BHASKAR PAI,

Patents Officer.

Council of Scientific & Industrial Research.

Dated this 18th day of August 1966.

MGIPS(KSR)—S14—3 Patent/69—23-5-75—100.