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"IMPROVEMENTS IN OR RELATING TO PROCESS FOR THE PRODUCTION OF NEGATIVE ACTIVE MATERIAL FOR POCKET TYPE AND PRESSED MASS TYPE NICKEL CADMIUM CELLS."

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

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This invention relates to a process for the preparation of negative active material for production of pocket type and pressed mass type nickel-cadmium cells.

Hitherto it has been proposed to precipitate ^{electrolytically} the negative active material from cadmium salts such as nitrate, sulphate, chloride of the respective metal under certain conditions which are not very well described. Further the screen analysis and activity depend very much on the conditions of precipitation.

The active materials prepared as per conditions in literature are not active enough to give the required capacity, and also the conditions do not produce reproducible results.

The object of the invention is to obviate these disadvantages by developing the process for the production of active material for pocket type and pressed mass type nickel-cadmium cells under standardised conditions to produce a highly active mass by incorporation of certain addition agents such as iron salts so that the coefficient of utilisation is better than that obtained in usual practice.

Optimum conditions have been worked out to produce negative active material.

According to the present invention, there is provided a process for the production of active material for use in pocket type and pressed mass type nickel cadmium cells by preparing negative active material by adding iron oxide to cadmium hydroxide characterised in that the negative active material is prepared by (a) ^{chemically} precipitating cadmium hydroxide and iron oxide/hydroxide from a solution containing cadmium and iron salts or (b) precipitating

iron oxide/hydroxide separately and adding to cadmium hydroxide precipitate.

Thus, the resulting negative active material for use in pocket type and pressed mass type nickel cadmium cells comprises cadmium hydroxide and iron oxide/hydroxide co-precipitated from a solution containing cadmium and iron salt.

The cadmium hydroxide is precipitated from a solution of one or more of the cadmium salts such as cadmium nitrate, cadmium sulphate, cadmium chloride or any suitable cadmium salt preferably the cadmium nitrate to which a nitrate, sulphate, chloride or any suitable salt of iron is added.

The concentration of cadmium salt is kept between 0.1 and 1.25 g/ml, preferably 0.5 g/ml and the concentration of iron salt added is kept between 15% and 65% preferably 40% by weight of the cadmium salt.

Sodium and/or potassium hydroxide is used for the precipitation of cadmium hydroxide and iron oxide/hydroxide for the negative active material.

The concentration of sodium and/or potassium hydroxide for the precipitation of cadmium hydroxide and iron oxide/hydroxide is kept between 30% and 70% preferably 69% by weight.

The precipitation is carried out at a temperature between 20°C and 55°C preferably at 35°C for negative active material.

The sodium and/or potassium hydroxide solution is added at a constant rate with vigorous stirring, in a duration of 2 to 30 minutes preferably in 10 minutes.

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Alternatively, an addition of a powder of iron oxide/hydroxide is also made to the cadmium hydroxide to give the advantages of co-precipitation.

The addition of iron oxide/hydroxide to cadmium hydroxide ranges from 20% to 65% preferably 50% by weight of cadmium hydroxide.

The particle size of iron oxide is kept between 106 to 150 microns.

PREPARATION OF NEGATIVE ACTIVE MATERIAL

Preparation of cadmium hydroxide:

Very pure cadmium nitrate [$\text{Cd}(\text{NO}_3)_2 \cdot 4 \text{H}_2\text{O}$] conforming to Analar grade of not less than 99.5% assay is used to prepare cadmium hydroxide precipitate.

Pure sodium hydroxide containing not less than 98% sodium hydroxide is used for precipitation.

420 gms of cadmium nitrate is dissolved in distilled water and was made upto 1100 ml of solution. 115 gms of sodium hydroxide or 160 gms of potassium hydroxide is taken and dissolved in distilled water and is made upto 170 ml or 230 ml in case of potassium hydroxide. The alkali solution is slowly added to the salt solution with constant stirring at the same time the temperature being maintained at 35°C with suitable cooling arrangement. The precipitate is washed well with distilled water repeatedly until the pH reaches 7. The precipitate is then dried at suitable temperature not exceeding 110°C for 16 hours. After drying, the precipitate is treated with hot distilled water to remove traces of nitrate. The final assay of the cadmium hydroxide is as shown below.

Cadmium	Not less than 76%
Copper	Less than 0.05%

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Mercury	Less than 0.05%
Zinc	Less than 0.05%
Nitrate as NaNO_3	Traces only

Optimum particle size distribution of the powder was as follows:

150 microns - 106 microns	65% to 95%
106 microns - 75 microns	5% to 35%
Less than 75 microns	Negligible

To this cadmium hydroxide powder, is added iron oxide of 106 to 150 microns size and of purity of 99.5%. The iron oxide preparation is given below.

Pure ferric nitrate is used to prepare iron oxide pure sodium hydroxide containing not less than 98% sodium hydroxide is used for precipitation.

400 gms of ferric nitrate is dissolved in distilled water and made upto 1000 ml. 130 gms of sodium hydroxide dissolved in distilled water and is made upto 250 ml.

Alkali solution is added to iron salt solution by constant stirring and the temperature is kept below 35°C by suitable cooling arrangement. The precipitate is washed well with distilled water to remove alkali till the pH was 7. The precipitate is then treated with hot distilled water to remove traces of nitrate and is dried. The precipitate is then roasted in an oven at 250°C-300°C to give iron oxide.

The final negative active material composition contains between 10% and 60% iron oxide. The powders are mixed in a suitable mixing machine.

The present invention consists of a process for preparation of negative active material for pocket type

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and pressed mass type nickel cadmium cells. The negative active material is a mixture of cadmium hydroxide and iron oxide.

The new elements in the process are inclusion of iron oxide/hydroxide for negative active material with material combination of cadmium hydroxide. Whereas the conventional processes for the production of active material do not include the inclusion of iron oxide/hydroxide to the negative active material.

EXAMPLE-I

Incorporating the negative active material prepared as described above, pellets are prepared for the assembly of the cell. Preparation of pellets is described below:

4 gms of the negative active material (cadmium hydroxide $[Cd(OH)_2]$, containing 30% by weight of iron oxide) is weighed. To this is added one gm. of electrolyte (30% KOH + 0.25% LiOH) and is made into a paste. The paste is pressed at a pressure of 5 tons per square inch to give a pellet of final dimensions 3.0 cm dia x 0.25 cm thick and is enclosed in a stainless steel wire mesh of 200 mesh size (BSS).

A suitable positive pellet is prepared out of nickel hydroxide active material.

The positive and negative pellets are then soaked in an electrolyte of 30% potassium hydroxide containing 0.25% lithium hydroxide for 12 hours.

The resultant pellets are then assembled into a completely sealed cell with two folds of nylon felt separator in nickel plated mild steel container. Number of cells are assembled as above and on charge discharge the cells gave a capacity of 200 milli ampere hours at 1 hour rate.

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EXAMPLE-III

Incorporating the negative active material prepared as described above, pockets were prepared for the assembly of the cell. Preparation of pockets is described below:

2.25 gms of negative active material (cadmium hydroxide containing 50% by weight of iron oxide) is mixed with 0.3 gms of electrolyte (30% KOH + 0.25% LiOH) to make a paste. This is pressed into a pellet and is introduced inside pockets having dimensions 4.0 cm x 1.5 cm. Four such pockets were mounted in a nickel plated mild steel frame, welding the ends to the frame. This constitutes one negative plate. The thickness of the negative plate is 0.225 cm.

Two negative plates are welded to a terminal post which constitutes a complete negative element.

A suitable positive plate prepared out of nickel hydroxide and welded to a terminal post constitutes positive element. The cell is assembled by combining negative and positive groups and punched PVC sheet is used as separator.

The plates are soaked in an aqueous solution of 30% potassium hydroxide containing 0.25% lithium hydroxide by weight for a period of 12 hours before being charged. The cell gave a capacity output of 1.5 A.hrs. at 5 hr rate.

The main advantages of the inventions are:

- 1) Methods for the preparation of active material for negative electrode has been standardised for producing pocket type and pressed mass type nickel cadmium cells indigenously.

(ii) The coefficient of utilisation of the active material prepared is around 30% which is higher than the usually reported values of 20%-25% in literature.

(iii) The addition of iron oxide not only enhances and stabilises the potential of the electrodes but it also reduces the quantity of cadmium hydroxide used and thereby brings down the cost of the cell.

The negative active material for use in pocket type and pressed mass type nickel cadmium cells comprises mainly a mixture of cadmium hydroxide and iron oxide. The conditions of precipitation have been standardised so that the particle size and the particle size distribution are within the optimum range. The activity and the coefficient of utilisation are maximum.

We claim:

1. A process for the production of negative active material for use in pocket type and pressed mass type nickel cadmium cells by preparing cadmium hydroxide active material by adding iron oxide and further characterised in that the negative active material is prepared by (a) *Chemically* precipitating cadmium hydroxide and iron oxide/hydroxide from a solution containing cadmium and iron salts or (b) precipitating iron oxide/hydroxide separately and adding to cadmium hydroxide precipitate.

2. A process as claimed in claim 1 wherein the cadmium hydroxide is precipitated from a solution of one or more of the cadmium salts such as cadmium nitrate, cadmium sulphate, cadmium chloride or any suitable cadmium salt preferably the cadmium nitrate to which a nitrate, sulphate, or chloride or any suitable salt of iron is added.

3. A process as claimed in claim (2) wherein the concentration of cadmium salt is kept between 0.1 and 1.25 g/ml, preferably 0.5 g/ml and the concentration of iron salt added is kept between 15% and 65% preferably 40% by weight of the cadmium salt.

4. A process as claimed in any of the preceding claims wherein sodium and/or potassium hydroxide is used for the precipitation of cadmium hydroxide and iron oxide/hydroxide for the negative active material.

5. A process as claimed in claim (4) wherein the concentration of sodium and/or potassium hydroxide for the precipitation of cadmium hydroxide and iron oxide/hydroxide is kept between 30% and 70% preferably 69% by weight.

6. A process as claimed in claim (5) wherein the precipitation is carried out at a temperature between 20°C and 55°C preferably 35°C.

7. A process as claimed in claim (6) wherein the sodium and/or potassium hydroxide solution is added at a constant rate with vigorous stirring, in a duration of 2 to 30 minutes preferably in 10 minutes.

8. A process as claimed in claim (1) wherein the addition of iron oxide/hydroxide to cadmium hydroxide ranges from 20% to 65% preferably 50% by weight of cadmium hydroxide.

9. A process as claimed in claim (8) wherein the particle size of iron oxide added is between 106 microns to 150 microns size.

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10. A process for the production of negative active material for use in pocket type and pressed mass type nickel cadmium cells substantially as hereinbefore described.

Dated this 2nd day of June, 1975

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