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PROVISIONAL SPECIFICATION

IMPROVEMENTS IN OR RELATING TO THE FORMATION OF PLATES FOR NICKEL CADMIUM BATTERIES.

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, Director, PENNAGARAM VYASA RAO VASUDEVA RAO, Scientist, THIRUMAL RAO VASANTHI, Senior Laboratory Assistant, all of the Central Electro-chemical Research Institute, Karaikudi-3, tamil Nadu, India, all Indian citizens.

The following Specification describes the nature of this invention.

This invention relates to improvements in or relating to impregnation and forming of plates for nickel cadmium batteries.

Hitherto it has been proposed to impregnate sintered matrices of nickel powder with nickel nitrate or cadmium nitrate or suitable salt solutions of either cadmium or nickel for either negative or positive as the case may be. To improve the process of impregnation and forming the active material, several other methods like thermal impregnation and electrolytic methods have been developed, but there is no reference to improve the conventional process by making certain additions, either to the impregnating solutions, or to the forming bath.

The object of this invention is to make certain additions to the impregnating solution to improve the process of precipitating the active material, inside the pores of the sintered matrices with a view to improve the efficiency of the active material.

To these ends the invention broadly consists in adding certain surface active agents to the impregnating solution or to the forming bath, in quantities varying between 0.001% to 0.2% by weight per litre of the impregnating solution, the quantity being limited by the solubility of the surfactant Cationic surfactants belonging to tetra substituted quaternary ammonium compounds with the general electronic configuration

$$\left[R : \overset{R_2}{\underset{R_4}{N}} : R_3 \right]^{+} X^{-}$$ in which N-R, N-R2 etc.

represents a covalent nitrogen-carbon bond; i. e. one of the groups R1—R4 is a hydrogen atom. X represents the anion. For example, the following surfactants like alkyl-di-methyl benzyl-ammonium-chloride, cetyl trimethyl ammonium bromide, chloride, cetyl triethyl ammonium iodide, lauryl dimethyl benzyl ammonium bromide, p. steryl phenyltrimethyl ammonium metho sulphate.

The suitable weight of the compound is dissolved in a small quantity of the impregnating solution first, heat applied, if necessary to dissolve it completely, and then added to the main bulk of the solution. The impregnation is carrid out in vacuum by the usual process. The formation is carried out electrolytically with sodium hydroxide of 30% by weight, at normal temperature. The impregnated plaques are cathodically polarised at a current density of 5 to 20 amps/dm^2. The post-treatment of the impregnated plaque is carried out in the usual way.

The following typical examples are given to illustrate the invention.

Example I

POSITIVE PLATES

Impregnating solution :	Nickel nitrate
Addition agent :	Cetyl trimethyl ammonium ioxide—0.05% by weight/ letre of impregnating solution
Forming solution.	30% sodium hydroxide
Current density.	10 amps/dm^2
Time of formation :	½-an-hour

Plates formed as above were compared with plates formed without the addition agent and gave the following performance (an average results of 3 different plates in each)

	Percentage of utilisation at			
	C/5 rate	C rate	5C rate	10C rate
Plates formed with addition agent	93%	75%	74%	49%
Plates formed without addition agent	82%	65%	46%	23%

Example II

NEGATIVE PLATES

Impregnation solution :	Cadmium nitrate
Addition agent.	Cetyl triethyl ammonium bromide-0.05% by weight/ litre of impregnating solution
Forming solution :	30% sodium hydroxide
Current density :	10 amps/dm^2
Time of formation :	½-an-hour

Plates formed as above were compared with plates formed without addition agent gave the following performance (an average of 2 different plates in each)

	Percentage of utilisation at			
	5 hr rate	1C rate	5C rate	10C rate
Plates formed with addition agent	90%	70%	30%	22%
Plates formed without addition agent	80%	52%	15%	13%

Price : TWO RUPEES

The main advantages of the invention are :

1) Increase in parcentage of utilisation :

2) Consequently, the weight of the active material required to give the specified capacity will also be lower. This will reduce the total cost Since nickel is saved, this will also reduce the dependence on the import of the same.

3) Similarly, a saving in cadmium consumption is also achieved.

Sd/- R. Bhaskar Pai
Patent Officer
Council of Scientific and Industrial Research

Dated this 28th day of June, 1972

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, Director, PENNAGARAM VYASA RAO VASUDEVA RAO, Scientist, THIRUMAL RAO VASANTHI, Senior Laboratory Assistant, all of the Central Electro-chemical Research Institute, Karaikudi-3, Tamil Nadu, India, all 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 impregnation and forming of plates for nickel cadmium batteries.

Hitherto it has been proposed to impregnate sintered matrices of nickel powder with nickel nitrate or cadmium nitrate or suitable salt solutions of either cadmium or nickel for either negative or positive as the case may be. To improve the process of impregnation and forming the active material, several other methods like thermal impregnation and electrolytic methods have been developed, but there is no reference to improve the conventional process by making certain additions either to the impregnating solutions, or to the forming bath.

The conventional methods and the recent methods of thermal impregnation and direct formation methods aims at reducing the number of impregnating cycles but not improving the efficiency of utilisation. The present method aims at reducing the number of impregnating cycles as well as the efficiency of utilisation as compared in the examples given.

The object of this invention is to make certain additions to the impregnating solution to improve the process of precipitating the active material, inside the pores of the sintered matrices, with a view to improving the efficiency of the active material. This improved efficiency also saves the total quantity of active material required for a given capacity.

The new finding consists in making certain additions to the impregnating solution and forming the same into the active material which has got a higher efficiency of utilisation.

The new result obtained by the addition of certain quarternary ammonium compounds having a configuration of

$$R_1 : \overset{R_3}{\underset{R_4}{N}} : R_2 \quad X^-$$

in which N-R, N-R_3 etc. represents a covalent nitrogen—carbon bond, i.e. one of the groups R_1 .....R_4

is a alkyl or aryl substituent for a hydrogen atom X represents the anion. For example the following surfactants like alkyl-di-methyl benzyl ammonium chloride, cetyl trimethyl ammonium bromide/chloride, cetyl tri-ethyl ammonium iodide, lauryl-di-methyl-benzyl ammonium bromide, P. steryl phenyl tri-methyl ammonium metho sulphate, is illustrated in the examples given

It can be seen that a large difference in the utilization efficiency is obtained with the addition agent especially at high rates Such a change brought about by an addition agent to the impregnating solution has not been reported earlier

The present invention consists of addition of certain quarternary ammonium compounds to the impregnating solution used for preparing nickel cadmium battery plates The active material produced with such an addition has a higher utilisation efficiency than the active material obtained by the conventional impregnation and forming method. This increased efficiency is more pronounced at high rates of discharge than at normal rates.

According to the present invention, there is provided an improved process for producing positive and negative plates of alkaline nickel cadmium batteries which consists in adding a quarternary ammonium compound of the general formula

$$R_1 , \overset{R_3}{\underset{R_4}{N}} : R_2 \quad X^-$$

to an impregnating solution consisting of nickel or cadmium salts as the case may be, and impregnating the solution into porous sintered matrices in vacuum and precipitating and forming the nickel or cadmium hydroxide electrolytically in a solution of sodium hydroxide.

The process of impregnation is carried out in vacuum by keeping sintered matrices in an aqueous solution of nickel nitrate or cadmium nitrate for the positive or the negative respectively.

A quarterinary ammonium compound with the configuration,

$$R_1 : \overset{R_3}{\underset{R_4}{N}} R_2 \quad X^-$$

for example alkyl dimethyl-benzyl-ammonium chloride, cetyltrimethyl ammonium iodide or hydroxide, cetyl trimethyl ammonium bromide chloride or hydroxide, dimethyl-benzyl ammonium bromide, P-phenyl-tri-methyl ammonium metho sulphate and in particular cetyl trimethyl ammonium bromide is added alone, or in combination, to the impregnating solution.

The concentration of quarterinary ammonium compound ranges between .01 to .5% wt/v.

After impregnation, the active materials of nickel hydroxide for the positive and cadmium hydroxide for

the negative are precipitated inside the porse of the sintered matrices by dipping in an aqueous solution of sodium hydroxide, and electrochemically treating the same.

The impregnated plates are treated as cathodes.

A current density ranging between 2 to 20A per dm^2 preferably 10 amp/dm^2 is used, at the temperature between 30 to 90°C preferably at 40°C.

Thus, the process of impregnation is carried out by keeping sintered matrices in an aqueous solution of nickel nitrate or cadminm nitrate for the positive or the negative respectively and evacuating the system.

Typical examples

POSITIVES

	Percentage of utilisation at			
	C/5 rate	1C rate	5C rate	10C rate
Plates formed with addition agent.	93%	75%	74%	49%
Plates formed without addition agent	82%	65%	46%	23%

	Percentage of utilisation at C/5 rate at 0°C
Plates formed with addition agent	87.9%
Plates formed without addition agent	67,11%

NEGATIVES

	Percentage of utilisation at			
	C/5 rate	1C rate	5C rate	10C rate
Plates formed with addition agent	90%	70%	30%	22%
Plates formed without addition agent	80%	52%	15%	13%

	Percentage of utilisation at C/5 rate at 0°C
Plates formed with addition agent	67%
Plates formed without addition agent	43%

The main advantages of the invention are (1) increase in percentage of utilisation (2) consequently a lower requirement of the active material to give the specified capacity. This will reduce the total cost. Since nickel is saved, this will also reduce the dependence on the import of the same. (3) a saving in cadmium cosnumption is also achieved.

Sintered matrices for nickel cadmium battery are usually impregnated with the solution of $Ni(NO_3)_2$ for the positive and the solution $Cd(NO_3)_2$ for the negatives. Several cycles of impregnation and forming are required for getting the proper quantity of active material impregnated and the efficiency of utilisation of such an active material is low. It has been found that making an addition of certain quarternary ammonium compounds already mentioned above improves the efficiency of the active material so that the quantity of active material required to produce the given capacity is less and so more economical. The performance is more markedly improved at high rates of discharge and at low temperatures. The machanism of such an improvement is not well understood though the main reason appears to be reduction in the size and distribution of the particles of the active material, which increases the total surface area.

WE CLAIM :—

1. An improved process for producing positive and negative plates of alkaline nickel cadmium batteries comprises adding a quaterinary ammonium compound of the general formula

$$R_1 : \underset{R_4}{\overset{R_2}{N}} ; R_3 \quad X^-$$

to an impregnating solution consisting of nickel or cadmium salts as the case may be, and impregnating the solution into porpous sintered matrices in vacuum and precipitating and forming the nickel or cadmium hydroxide electrolytically in a solution of sodium hydroxide.

2. A process as claimed in Claim 1 where the process impregnation is carried out in vacuum by keeping sintared matrices in an aqueous solution of nickel nitrate or cadmium nitrate for the positive or the negative respectively.

3. A process as claimed in claim 1 or to wherein a quarterinary ammonium compound with the configuration.

$$R_1 : \underset{R_4}{\overset{R_2}{N}} : R_3 \quad X^-$$

for example alkyl dimethyl-benzyl-ammonium chloride, Cetyl-trimethyl ammonium iodide or hydroxide, cetyl trimethyl ammonium bromide chloride or hydroxide, dimethyl-benzyl ammonium bromide, P-phenyl-trimethyl-ammonium method sulphate and in particular cetyl trimethyl ammonium bromide is added alone, or in combination, to the impregnating solution

4. A process as claimed in any of the preceding claims wherein the concentration, of quarterinary ammonium compound ranges between .01 to .5% wt/y.

5. A process as claimed in any of the preceding claims wherein after impregnation, the active materials of nickel hydroxide for the positive and cadmium hydroxide for the negative are precipitated inside the pores of the sintered matrices by dipping in an aqueous solution of sodium hydroxide, and electrochemically treating the same.

6. A process as claimed in any of the previous claim wherein the impregnated plates are treated as cathodes.

7. A. process as claimed in any of the preceding claims wherein a current density ranging between 2 to 20A per dm^2 preferably 10 amp/dm^2 is used, at the temperature between 30 to 90°C preferably at 40°C.

8. An improved process for producing positive and negative plates of alkaline nickel cadmium batteries substantially as herein before described.

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