

SPECIFICATION

Preparation of nickel(II) hydroxide

5 The present invention relates to a process for preparing nickel hydroxide as powder for use as the active constituent of the positive electrode mass in alkaline electrochemical cells by reaction of a nickel (II) salt with alkali hydroxide.

10 $\text{Ni}(\text{OH})_2$ for use as the active constituent of the positive mass in alkaline cells is prepared at the present time by a protracted and complicated precipitation process. This process involves precipitating the hydroxide from nickel(II) salt solutions by means of alkali hydroxides, separating the precipitate from the mother liquor, and washing and drying the precipitate. On account of the poor filtrability of the precipitation product, which is initially present in suspended form, the process is very time-consuming, and in some cases lasts several days.

20 In contrast, the application of nickel hydroxide as an active mass to a highly porous sintered electrode framework or plate, which similarly involves a precipitation process, can be carried out quickly. In this connection, the sintered electrode framework or plate is dipped in a highly concentrated nickel nitrate solution in order to fill the pores. The impregnated plate is next treated with concentrated alkali hydroxide in order to precipitate nickel hydroxide, and is then washed and dried. The whole process lasts less than one hour and provides a highly utilisable incorporated mass.

30 An object of the present invention is to provide a process for the efficient production of $\text{Ni}(\text{OH})_2$ for use as the active constituent of a positive electrode mass in an alkaline electrochemical cell, the process enabling the $\text{Ni}(\text{OH})_2$ to be prepared from a water-soluble nickel salt without the encumbrance of large amounts of liquid and without the otherwise necessary separation of the reaction product by a time-consuming filtration.

40 In accordance with the present invention there is provided a process for preparing nickel hydroxide for use as the active constituent of positive electrode masses in alkaline electrochemical cells, comprising reacting a water-soluble nickel(II) salt with alkali hydroxide, wherein the nickel(II) salt is pulverulent and is converted directly to $\text{Ni}(\text{OH})_2$ by highly concentrated solution of the alkali hydroxide whereby a homogeneous solution phase of Ni^{2+} ions is suppressed.

50 Preferably the water-soluble nickel(II) salt is nickel nitrate ($\text{Ni}(\text{NO}_3)_2$) and the alkali hydroxide is sodium hydroxide.

55 The pulverulent water-soluble nickel(II) salt (e.g. $\text{Ni}(\text{NO}_3)_2$) may be provided in granular, powder or other particulate form.

60 In carrying out the process of the invention preferably $\text{Ni}(\text{OH})_2$ is formed in an almost topochemical reaction, i.e. with the virtual exclusion of the solution phase. The information of the nickel nitrate particles (or other water-soluble nickel(II) salt) is contained in the $\text{Ni}(\text{OH})_2$ in the sense that the latter is similar to the nickel nitrate (or other nickel salt) as regards morphology and particle size.

70 Such a conversion is possible since the reactants are preferably not added to one another directly but in counter-current. If pure nickel nitrate powder is primarily contacted with less concentrated hydroxide, no complete conversion to $\text{Ni}(\text{OH})_2$ takes place. Rather, intermediately basic nickel nitrates, for example of the composition $\text{Ni}(\text{NO}_3)_2 \cdot \text{Ni}(\text{OH})_2 \cdot 6\text{H}_2\text{O}$, are formed. With increasing proportions of $\text{Ni}(\text{OH})_2$ in the molecule, the water solubility of the basic nickel nitrates decreases. Also, a considerable amount of water from the hydroxide solution is bound as water of crystallisation, the precipitation is restricted to the reaction site, and the formation of a suspension is largely avoided.

80 If the basic nickel nitrates come into contact with unconsumed highly concentrated hydroxide, i.e. a hydroxide with a low water content, complete conversion to $\text{Ni}(\text{OH})_2$ is brought about.

85 The precipitation therefore preferably takes place in such a way that nickel nitrate (or other water-soluble nickel(II) salt) powder is moved along a conveying path, and that highly concentrated hydroxide moves from one end of this conveying path in counter-current to the stream of powder, whereby the hydroxide concentration falls as a result of the conversion with the powder and only a moderately concentrated hydroxide is present at the point of application of the fresh nickel nitrate (or other water-soluble nickel(II) salt). In this connection, the amount of conveyed hydroxide is kept so low that the reaction mixture is at best well moistened, but no suspension is formed. Furthermore, the hydroxide at the end of its conveyance path must not be so depleted in OH^- ions that dissolution of fresh $\text{Ni}(\text{NO}_3)_2$ (or other water-soluble nickel (II) salt) takes place before the precipitation of basic nitrates or of $\text{Ni}(\text{OH})_2$, i.e. the precipitation of the $\text{Ni}(\text{OH})_2$ must occur in situ.

100 The alkali hydroxide solution is selected such that the entire amount of water (including the water of crystallization of the nickel salt) is sufficient to keep the reaction product in solution. For example, if using nickel nitrate, the sodium hydroxide solution should have a concentration smaller than 16 N, as otherwise there would not be sufficient water available.

105 The whole precipitation process need not necessarily be carried out in a continuous manner, and a procedure involving several discrete stages is also possible. The expression "in countercurrent" simply refers to the direction in which the reactants nickel salt and alkali hydroxide are conveyed relative to one another.

110 The accompanying drawing shows diagrammatically by way of example apparatus used in carrying out a process according to the invention.

115 Referring to the drawing $\text{Ni}(\text{NO}_3)_2$ powder is added, preferably by a metering device (not shown), to the lower end of an inclined, upwardly moving run of a conveyor belt 1. Sodium hydroxide that has been added at the upper end of the run flows countercurrent to the upwardly moving powder. During its downward path the hydroxide comes into contact with the oppositely moving basic nickel nitrate 2, in which the proportion of $\text{Ni}(\text{NO}_3)_2$ not yet converted

to $\text{Ni}(\text{OH})_2$ predominates. The result is a depletion of the hydroxide of OH^- ions. For example, the fresh sodium hydroxide at the higher located point of application to the conveyor belt is 11 N while at the lower end it is only 2 N.

In order to prevent the $\text{Ni}(\text{NO}_3)_2$ sliding off the conveyor belt and to ensure a certain time of contact with the hydroxide, trough-like depressions 3 are provided in the conveyor belt transverse to the direction of movement. The hydroxide thus flows in a cascade-like manner down over the edges of the depression, a new reaction between the hydroxide and the contents of the depressions taking place with each step. The basic nature of the nickel nitrate thus increases with every depression during its passage from the bottom to the top of the run of the conveyor belt, until $\text{Ni}(\text{OH})_2$ free of $\text{Ni}(\text{NO}_3)_2$ flows off the conveyor belt at 4 when the belt changes direction.

The hydroxide flowing off at 5 may, after removal of entrained alkali nitrate, be passed to a vacuum evaporator and recycled to the process after concentration. The washing out of the nitrate from the $\text{Ni}(\text{OH})_2$ leaving at 4 presents no problems on account of its large crystalline nature, in contrast to the voluminous, flaky product obtained in the conventional precipitation.

Since $\text{Ni}(\text{OH})_2$ has no intrinsic conductivity, it may in certain cases be of interest to convert the active mass (i.e. the $\text{Ni}(\text{OH})_2$) wholly or partially into higher grade nickel oxides with a chemical oxidising agent such as potassium peroxydisulphate ($\text{K}_2\text{S}_2\text{O}_8$). In this way the electrodes, preformed for incorporation into a cell can be given a certain electron conduction.

A partial oxidation of the $\text{Ni}(\text{OH})_2$ by means of $\text{K}_2\text{S}_2\text{O}_8$ may also be effected with particular advantage by the process of the invention by converting the pulverulent nickel nitrate with a mixture of pure sodium hydroxide and potassium peroxydisulphate instead of with pure sodium hydroxide alone.

CLAIMS

1. A process for preparing nickel hydroxide for use as the active constituent of positive electrode masses in alkaline electrochemical cells, comprising reacting a water-soluble nickel(II) salt with alkali hydroxide, wherein the nickel(II) salt is pulverulent and is converted directly to $\text{Ni}(\text{OH})_2$ by highly concentrated solution of the alkali hydroxide, whereby a homogeneous solution phase of Ni^{2+} ions is suppressed.

2. A process according to claim 1, wherein the pulverulent nickel(II) salt is $\text{Ni}(\text{NO}_3)_2$.

3. A process according to claim 1 or 2, wherein the alkali is NaOH .

4. A process according to any of claims 1 to 3, wherein the solution of alkali contains a chemical oxidising agent.

5. A process according to claim 4, wherein the oxidising agent is potassium peroxydisulphate.

6. A process according to any preceding claim, wherein the highly concentrated solution of alkali hydroxide is such that the entire amount of water (including the water of crystallization of the nickel salt) is sufficient to keep the reaction product in solution.

7. A process according to any preceding claim, wherein the alkali solution flows in countercurrent to the water-soluble nickel(II) salt.

8. A process according to any preceding claim, wherein the water-soluble nickel(II) salt is in granular or powder form.

9. A process for preparing nickel hydroxide for use as the active constituent of positive electrode masses in alkaline electrochemical cells, substantially as described herein with reference to the accompanying drawing.

10. A process according to claim 1, substantially as described herein with reference to the accompanying drawing.

11. An alkaline electrochemical cell comprising a positive electrode mass having as active constituent nickel hydroxide prepared by a process according to any preceding claim.

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10 thus increases with every depression during its passage from the bottom to the top of the run of the conveyor belt, until $\text{Ni}(\text{OH})_2$ free of $\text{Ni}(\text{NO}_3)_2$ flows off the conveyor belt at 4 when the belt changes direction.

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30 mass (i.e. the $\text{Ni}(\text{OH})_2$) wholly or partially into higher grade nickel oxides with a chemical oxidising agent such as potassium peroxydisulphate ($\text{K}_2\text{S}_2\text{O}_8$). In this way the electrodes, preformed for incorporation into a cell can be given a certain electron conduction.

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CLAIMS

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50 2. A process according to claim 1, wherein the pulverulent nickel(II) salt is $\text{Ni}(\text{NO}_3)_2$.

3. A process according to claim 1 or 2, wherein the alkali is NaOH .

55 4. A process according to any of claims 1 to 3, wherein the solution of alkali contains a chemical oxidising agent.

5. A process according to claim 4, wherein the oxidising agent is potassium peroxydisulphate.

60 6. A process according to any preceding claim, wherein the highly concentrated solution of alkali hydroxide is such that the entire amount of water (including the water of crystallization of the nickel salt) is sufficient to keep the reaction product in solution.

7. A process according to any preceding claim, wherein the alkali solution flows in countercurrent to the water-soluble nickel(II) salt.

8. A process according to any preceding claim, wherein the water-soluble nickel(II) salt is in granular or powder form.

9. A process for preparing nickel hydroxide for use as the active constituent of positive electrode masses in alkaline electrochemical cells, substantially as described herein with reference to the accompanying drawing.

10. A process according to claim 1, substantially as described herein with reference to the accompanying drawing.

80 11. An alkaline electrochemical cell comprising a positive electrode mass having as active constituent nickel hydroxide prepared by a process according to any preceding claim.

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