

1

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**METHOD OF MANUFACTURING PURE NICKEL HYDROXIDE**

Charles Cuenot, Sainte-Suzanne, France, assignor to Le Nickel, societe anonyme, Paris, France, a French company

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The present invention relates to a novel method of manufacturing nickel hydroxide by an electrochemical process whereby this substance can be obtained directly from nickel mat (preferably refined by a metallurgical process) and from which any impurities detrimental to its subsequent applications (notably as an additive to nickel-plating baths) have been removed.

According to the techniques commonly applied in this field, high-purity nickel hydroxide is obtained from nickel sulfate or nickel chloride by lime or soda transfer, the salts, sulfate or chloride themselves being obtained beforehand according to conventional methods notably from pure nickel or nickel oxide. In this last case, pure nickel or nickel oxide may be prepared from nickel mat by applying various known refining processes, whether of the dry or moist type.

With this invention it is possible to produce high-purity nickel hydroxide directly from nickel mat, this possibility being economically advantageous in comparison with conventional methods in that it simplifies very considerably the manufacturing process.

This invention is concerned with a novel method whereby the greater part of chlorine residues or other anionic elements (such as sulfate, etc.), may be removed from the nickel hydroxides in the proportions to be set forth presently.

Moreover, this invention is concerned with a method capable of producing nickel hydroxides having the lowest possible contents of alkaline or alkaline-earth metal residues.

Other objects and advantages of this invention will appear as the following description proceeds.

As a rule, the present invention relates to a method of producing nickel hydroxide by an electrolytic process comprising the following steps:

(1) An anodic corrosion of nickel-mat anodes by exerting a direct electrolytic action in the anode compartments of an electrolysis cell divided into compartments and containing as the electrolyte an aqueous solution of a water-soluble chloride or sulfate of an alkaline or alkaline-earth metal such as notably sodium, potassium, magnesium, calcium, to produce a high nickel-chloride, or nickel-sulfate, anolyte in an acid medium having a pH value of 2 to 4.

(2) Diffusing the anolyte towards the cathode compartment of said electrolytic cell through fabric partitions disposed between the anode and cathode compartments.

(3) Precipitating the nickel into the cathode compartments in the form of nickel hydroxide in a basic medium having a pH value of 8 to 10.

(4) Continuously extracting from the electrolysis cell the nickel hydroxide suspended in the electrolyte.

(5) Decanting this suspension in a static decanter to concentrate the suspension from 4 to 6 grams of nickel per liter to 25–30 grams of nickel per liter.

(6) Repeatedly filtering the concentrated suspension while washing the nickel hydroxide with water on the filter, and effecting a repulping stirring, at the rate of three filtration stages and water washing on the filter, and two intermediate repulping steps.

2

Preferably, this method is carried out continuously.

The nickel-mat anodes comprise mainly nickel and sulfur, 76% to 83% by weight of nickel and 23% to 16% by weight of sulfur. The contents of elements other than nickel and sulfur lie within the following limits, by weight:

	Percent
Co -----	From 0.30 to 0.40.
Fe -----	From 0.04 to 0.08.
Cu -----	From 0.050 to 0.065.
Al <sub>2</sub> O <sub>3</sub> -----	From 0.01 to 0.02.
SiO <sub>2</sub> -----	From 0.020 to 0.025.
MgO -----	Traces.
Miscellaneous -----	According to the mat origin.
Insoluble -----	About 0.004.

The corrosion of the immersed portions of the anodes takes place regularly and uniformly. The dissolution is stopped when the current rises. Then, only a stump of anode (undissolved mat) and a sponge of sulfur remain. The sulfur sponge contains from 2% to 3% of the total initial nickel. These two wastes may be treated for on the one hand making new anodes by fusion and, on the other hand, if desired, recovering the sulfur and nickel separately.

A complete method of continuously manufacturing nickel hydroxide according to this invention is described hereinafter, by way of example, by using a sodium sulfate electrolyte.

Nickel-mat anodes are anodically corroded in anode compartments filled with a sodium sulfate, Na<sub>2</sub>SO<sub>4</sub> electrolyte, the Na<sub>2</sub>SO<sub>4</sub> concentration ranging from 100 to 150 g./liter. This electrolyte becomes charged with Ni to form a mixture of nickel sulfate and sodium sulfate. The nickel concentration maintained by continuous circulation ranges from 4 to 8 g./liter of nickel. The pH in the anode compartment is kept at a value of 2 to 4.

The electrolytic dissolution takes place at between 100 to 150 a./sq. m. of anode current density consistent with an adequate anode efficiency and the absence of oxygen release. A difference in level of 4 to 6 cm. is maintained between the anode and the cathode compartments by proper partitioning and the use of partition fabric elements, whereby the anolyte will flow continuously towards the cathode compartments.

The cathode compartment is kept at a pH value of 8 to 10 and the nickel sulfate is precipitated therein in the form of nickel hydroxide within these pH limit values.

The cathodes consist of stainless steel or nickel sheets and are the seat of a release of hydrogen.

The bath is kept at a temperature ranging from 45° to 55° C., and the cathode compartment is stirred by using compressed air to keep the hydroxide in suspension and continuously sweeping the cathodes to avoid any undesired or parasitic deposit.

The hydroxide suspension flowing continuously from the electrolytic cells is decanted in a static decanter in order to raise the nickel concentration to about 25–30 g./liter.

This suspension is subsequently filtered on a drum-type rotary filter and then washed over the filter. To obtain a hydroxide having the minimum sodium content from the electrolyte, three filtration steps are carried out and followed by a washing step and two intermediate repulping steps with water by using 8 to 10 volumes of water per volume of hydroxide. These repulping steps should be effected in a minimum time, of the order of half an hour, to ensure an efficient washing of the hydroxide, in a lukewarm medium, the same applying to the filtration steps.

A temperature of 45° to 55° C. is adequate for performing these steps.

3

The hydroxide manufactured according to the method of this invention by using a  $\text{Na}_2\text{SO}_4$  electrolyte and from mat anodes having the composition set forth hereinabove assays as follows (the percentages being by weight):

	Percent
Ni -----	58
Co -----	0.15 to 0.20
Fe -----	0.030 to 0.050
Cu -----	0.015 to 0.025
Cr -----	Nil
Pb -----	<0.002
Zn -----	<0.003
Na -----	<0.05
S (sulfide) -----	<0.002
Cl -----	<0.003

The present invention has been described hereinabove with particular reference to the electrolysis of mat anodes in a sodium sulfate electrolyte, but it applies as well to any electrolysis of mat anodes in any type of alkaline or alkaline-earth salt electrolyte providing a cathode basic medium having a pH value ranging from 8 to 10.

Although the present invention has been described with specific reference to particularly efficient or optimum conditions, it will be readily understood by anybody conversant with the art that variations and modifications may be contemplated without departing from the spirit and scope of the invention, as set forth in the appended claims.

What I claim is:

1. A method of manufacturing pure nickel hydroxide which comprises the steps of effecting the anodic corrosion of nickel mat anodes having the following contents by weight: nickel 76% to 83%, sulfur 23% to 16%, cobalt 0.30% to 0.40%, iron 0.04% to 0.08%, copper 0.050% to 0.065%, by direct electrolytic action in anode compartments of an electrolysis cell divided into compartments which contains as an electrolyte an aqueous solution of a water-soluble salt selected from the group consisting of the chlorides and sulfates of sodium, potassium, magnesium

4

and calcium, to form an anolyte having a high-nickel salt content selected from the group consisting of the chloride and sulfate, in an acid medium having a pH value ranging from 2 to 4, diffusing the anolyte towards the anode compartments of the electrolysis cell through fabric partitions separating the anode compartments from the cathode compartments, precipitating the nickel in the cathode compartments in the form of nickel hydroxide, in a basic medium having a pH value ranging from 8 to 10, continuously extracting from the electrolysis cell the nickel hydroxide suspension contained in the electrolyte, decanting said suspension in a static decanter for concentrating the suspension from 4 to 6 grams of nickel per liter to 25 to 30 grams of nickel per liter, and a series of filtrations of the concentrated suspension in conjunction with the washing of the nickel hydroxide with water and repulping stirring, said series comprising three stages of filtering and washing with water on the filter, and two intermediate repulping steps.

2. A method as set forth in claim 1, which is carried out continuously.

3. A method as set forth in claim 1, wherein said anolyte is an aqueous solution of sodium sulfate containing 100 to 150 grams of  $\text{Na}_2\text{SO}_4$  per liter and charged with 4 to 8 grams of nickel per liter, the resulting nickel hydroxide containing by weight 58% of nickel, less than 0.05% of sodium and less than 0.002% of sulfur.

4. A method as set forth in claim 3, which is carried out continuously.

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HOWARD S. WILLIAMS, *Primary Examiner*.

D. R. JORDAN, *Assistant Examiner*.