

$\alpha$ -Ni(OH)<sub>2</sub> obtained by template homogeneous precipitation exhibits high electrochemical activity in supercapacitors. The main disadvantage is the high energy consumption for maintaining a high temperature during synthesis. To reduce energy consumption, it is proposed to lower the synthesis temperature. In the study,  $\alpha$ -Ni(OH)<sub>2</sub> was obtained by the method of cold template homogeneous precipitation using Culminal C8465 (0.5 %) as a template for 6 months at  $t=20-35$  °C. The electrochemical characteristics of the sample were studied by cyclic voltammetry and galvanostatic charge-discharge cycling of a pasted binder-free electrode made without introducing an external binder in the supercapacitor mode. It was determined that low-crystalline  $\alpha$ -Ni(OH)<sub>2</sub> was formed, consisting of agglomerates of spherical particles. Low specific characteristics of nickel hydroxide were revealed at the beginning of cycling due to blocking of the active surface. It was shown that the specific capacity of the sample increased with further cycling due to the breakdown of aggregates into smaller particles; specific capacities of 80 F/g and 38 mA·h/g were obtained. However, the lack of binding properties of the template residues was revealed, resulting in a decrease in specific characteristics. It was concluded that it was necessary to introduce an external binder. A previously undescribed effect of a significant increase in the specific capacity during drying of an alkali-impregnated electrode caused by the disintegration of particle agglomerates during alkali carbonization (the maximum capacity is 135 F/g and 69 mA·h/g) was revealed. It was concluded that using the revealed effect of any nickel hydroxide samples obtained by various methods of bulk template synthesis was promising

**Keywords:** nickel hydroxide, template synthesis, cold homogeneous precipitation, supercapacitor

# SYNTHESIS OF Ni(OH)<sub>2</sub>, SUITABLE FOR SUPERCAPACITOR APPLICATION, BY THE COLD TEMPLATE HOMOGENEOUS PRECIPITATION METHOD

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## 1. Introduction

Supercapacitors (SC) are modern chemical current sources (CCS). SC are widely used to start various electric motors (for example, in electric vehicles and pumping units), as starter CCS in cars with internal combustion engines. SC are also used in uninterruptible power supplies for computers, medical equipment, and critical infrastructure facilities. The best characteristics are possessed by hybrid supercapacitors with high charge-discharge rates. As a result, on the Faraday electrode of the hybrid supercapacitor, the electrochemical reaction proceeds on the surface and in the thin surface layer of the active substance particles. Therefore, special requirements are imposed for the active substance [1, 2]. The substance of the Faraday electrode should mainly consist of particles with a high specific sur-

face, having a nano- and submicron size. Nickel hydroxide is widely used to make the Faraday electrode of hybrid supercapacitors. Ni(OH)<sub>2</sub> is used both individually [3], in the form of a nanoscale [4] or ultrafine powder [5], and in the form of a composite with carbon nanomaterials (graphene oxide [6], carbon nanotubes [7]).

A large number of methods for obtaining Ni(OH)<sub>2</sub> and layered double nickel hydroxides on its basis have been developed [8]. Chemical precipitation methods include direct synthesis (adding an alkali solution to a Ni<sup>2+</sup> salt solution) [9] and reverse synthesis (slowly adding a nickel salt solution to an alkali solution) [10, 11], as well as a method of high-temperature two-step synthesis [12] or sol-gel method [13]. For the synthesis of nickel hydroxides, electrochemical methods are also used [14, 15], including continuous production in a slit diaphragm electrolyzer [16, 17].

For nickel hydroxide, two structural modifications are described [18]:  $\beta$ -form ( $\text{Ni}(\text{OH})_2$ , crystal structure of brucite) and  $\alpha$ -form ( $3\text{Ni}(\text{OH})_2 \cdot 2\text{H}_2\text{O}$ , crystal structure of hydrotalcite).  $\beta$ - $\text{Ni}(\text{OH})_2$  has high cycling stability and is widely used in the positive electrode of alkaline batteries and supercapacitors.  $\alpha$ - $\text{Ni}(\text{OH})_2$ , compared to  $\beta$ - $\text{Ni}(\text{OH})_2$ , is much more electrochemically active and can be used more effectively in hybrid supercapacitors. Therefore, the development and optimization of methods for obtaining electrochemically highly active  $\alpha$ - $\text{Ni}(\text{OH})_2$  are urgent problems.

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## 2. Literature review and problem statement

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The method and conditions of synthesis directly determine the micro- and macrostructure of  $\text{Ni}(\text{OH})_2$  particles, which determines the electrochemical characteristics of nickel hydroxide. For effective use in supercapacitors,  $\text{Ni}(\text{OH})_2$  must have certain characteristics [19], in particular, the  $\alpha$ -structure with optimal crystallinity, submicron size [5], and nanosize [4, 20] particles.

During the formation of nickel hydroxide, the nucleation rate is much higher than the crystal growth rate. Because of this,  $\text{Ni}(\text{OH})_2$  is formed by a two-stage mechanism [21]: a very fast first stage of the formation of a primary amorphous particle and a slow second stage of crystallization (aging) of the primary particle. As a result, a hydrophilic matrix precipitate is formed containing a large amount of included mother liquor. During vacuum filtration, the precipitate particles are compressed, and during the subsequent drying, they are sintered. As a result, the particle size increases significantly and the specific surface area decreases. This can be prevented by using two strategies:

1) the use of synthesis methods with a reduced nucleation rate and an increased crystal growth rate;

2) introduction of special substances in the reaction solution to prevent aggregation and primary particle growth.

The first strategy is implemented using the homogeneous precipitation method [22]. The essence of the method consists in the formation of  $\text{OH}^-$  precipitating ions directly over the entire volume of the solution as a result of the reaction of thermal hydrolysis of substances containing amino groups (urea [23, 24], hexamethylenetetramine [25]). Homogeneous precipitation can be carried out from aqueous solutions, from solutions with mixed solvents [26], or non-aqueous solutions, for example, based on ionic liquids [27]. To obtain ultrafine or nanosized particles, homogeneous precipitation of  $\text{Ni}(\text{OH})_2$  is carried out at temperatures up to 150–180 °C. For the same purpose, microwave heating is used [28].

The second strategy involves the use of surfactants [29, 30] or templates. In the latter case, template synthesis is carried out, i.e., the formation of a substance in a matrix (template). As a rule, this method is used to form coatings, in particular electrochromic  $\text{Ni}(\text{OH})_2$  films [31], tripolyphosphate coating [32], or the direct formation of an electrode on the surface of nickel foam [33]. In this case, the resulting films have a composite structure similar to polymer composites [34], and there is no need to remove the template for them. In [35], the possibility of using water-soluble templates for the preparation of  $\text{Ni}(\text{OH})_2$  was shown.

The most promising is a combination of both strategies – the use of template homogeneous precipitation. A template for the synthesis of  $\text{Ni}(\text{OH})_2$  in an aqueous solution must

have certain characteristics – water solubility, chemical affinity for nickel compounds, and the ability to form a 3D matrix in the solution. PEG6000 is used as a water-soluble template [36]. Polyvinyl alcohol (PVA) is a promising template. PVA is used as an agent controlling porosity in the synthesis of mesoporous alumina [37], hydroxyapatite crystals (together with sodium dodecyl sulfate) [38]. PVA is used in the preparation of MFI zeolite [39]; the use of MgO to remove dyes from wastewater [40], to form single-layer [41, 42] and multilayer [43] hydroxide coatings, to obtain 3D-structured macroporous oxides and hierarchical zeolites [44], as well as to improve the adhesion of coatings on the ITO surface [45]. In [46], the high efficiency of using cellulose ether Culminol C8465 as a template was shown.

However, template synthesis has a significant drawback – during synthesis, the template remains in the resulting substance and must be removed. The complexity of template removal led to the emergence of a whole “template-free” direction, i.e., template-free synthesis of ultradispersed systems, for example, the preparation of calcium titanate [47] and zeolite ZSM-5 [48]. Template-free synthesis is used to obtain nanosized  $\text{Ni}(\text{OH})_2$  [49, 50]. When used in a supercapacitor, the presence of a template in the composition of the active substance can lead to partial blocking of the particle surface and other side effects. From this point of view, the template from the active substance, in particular,  $\text{Ni}(\text{OH})_2$ , must be removed as completely as possible. Various methods are used to remove templates and surfactants, including ozone oxidation [51]. However, in the manufacture of a pasted electrode of a supercapacitor, a binder is introduced into the composition to prevent shedding of the active mass [29]. But the binder is an electrochemically inert material, which reduces the specific capacity of the active mass. In [52], the negative effect of a high residual amount of template on the supercapacitor properties of  $\text{Ni}(\text{OH})_2$  was shown. However, it is promising to use another strategy – the use of a template as an internal binder at the stage of forming a pasted-type electrode. The possibility of implementing this strategy is shown in [53].

It is necessary to point out a significant drawback of the homogeneous precipitation method – the need to use high temperatures (80–95 °C) of synthesis for the hydrolysis of urea or other similar substance. Heating significantly increases the cost of  $\text{Ni}(\text{OH})_2$  and increases its crystallinity. Homogeneous precipitation of nickel hydroxide at room temperature is limited by the low rate of hydrolysis of urea (or similar compounds). However, research in this direction has not been carried out.

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## 3. The aim and objectives of the study

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The study aims to assess the possibility of performing cold template homogeneous precipitation and to study the characteristics of the resulting  $\text{Ni}(\text{OH})_2$ . This will reduce energy consumption during production.

To achieve the aim, the following objectives were set:

– visually reveal the course of the first proposed long-term synthesis of nickel hydroxide by the method of template homogeneous precipitation at room temperature and qualitatively evaluate the formed  $\text{Ni}(\text{OH})_2$ ;

– if the process of cold homogeneous precipitation is successful, quantitatively study the structural and electrochemical characteristics of the obtained nickel hydroxide to assess the prospects of the proposed method.

#### 4. Materials and methods for obtaining samples of nickel hydroxide and studying the characteristics

##### 4.1. Method of obtaining the main sample of nickel hydroxide

To synthesize the starting  $\text{Ni}(\text{OH})_2$ , we used the method of template homogeneous precipitation from a 1 M solution of nickel nitrate containing 1 M urea in the presence of a high molecular weight soluble template Culminal C8465 with a concentration of 0.5 wt %. In contrast to the method described in [53], the synthesis was carried out at room temperature (20–30 °C) for 6 months. After obtaining, the precipitate was filtered under vacuum, dried at 75 °C for a day, ground, soaked in distilled water (for 24 hours), filtered, and re-dried under the same conditions.

##### 4.2. Study of the characteristics of nickel hydroxide samples

To study the crystal structure of the obtained nickel hydroxide, we used X-ray phase analysis (XPA) using a DRON-3 X-ray diffractometer (Russia). XPA parameters:  $\text{Co-K}\alpha$  radiation in the  $2\theta$  range of 10–90° with a scan rate of 0.1°/s.

Scanning electron microscopy (SEM) was used to study the morphology of nickel hydroxide particles (electron microscope 106–I (SELMI, Ukraine)).

To study the electrochemical characteristics of synthesized  $\text{Ni}(\text{OH})_2$ , the following methods were used:

1) cyclic voltammetry. The study was carried out in the potential range of 0–500 mV (relative to the SCE), the sweep rate was 1 mV/s. When constructing voltammograms, the potentials were recalculated to NHE (normal hydrogen electrode);

2) galvanostatic charge-discharge cycling in the supercapacitor mode. Charge and discharge were performed at  $i=10, 20, 40, 80,$  and  $120 \text{ mA/cm}^2$ . At each current density, 5 charge-discharge cycles were carried out. The discharge curves were used to calculate the specific capacities  $C_{\text{sp}}$  (F/g) and  $Q_{\text{sp}}$  (mA·h/g). Charge-discharge cycling in the supercapacitor mode was carried out twice: primary – for a freshly made electrode and repeated – for the same electrode after drying and storage for 12 hours.

Both methods were carried out in a special electrochemical cell SEC-2 (USSR) using an electronic potentiostat-galvanostat Ellins R-8 (RF). For research, we made binder-free working electrodes (without the introduction of an external binder [52, 53]). The composition of the active mass: nickel hydroxide (84 wt %), GAK-2 graphite (16 wt %). A 6M KOH solution was used as the electrolyte. The Ni grid was used as a counter electrode. Ag/AgCl (saturated) was used as a reference electrode.

#### 5. Results of the study of the characteristics of nickel hydroxide obtained by cold template homogeneous precipitation

##### 5.1. Proceeding of long-term synthesis of nickel hydroxide

Visual observation revealed the passage of the first implemented template homogeneous precipitation. It was shown that the first particles of nickel hydroxide were formed 32 days after the start of the synthesis, which indicates the presence of an induction period. It was revealed

that, as a result of 6-month synthesis at room temperature, nickel hydroxide of an unusual type was formed – a fine crystalline precipitate of  $\text{Ni}(\text{OH})_2$ . It should be noted that, under normal conditions, the synthesized  $\text{Ni}(\text{OH})_2$  is a hydrophilic precipitate.

##### 5.2. Study of the characteristics of the obtained nickel hydroxide

The results of X-ray phase analysis (Fig. 1) show that the sample of nickel hydroxide obtained by cold template homogeneous precipitation is  $\alpha\text{-Ni}(\text{OH})_2$  with low crystallinity.

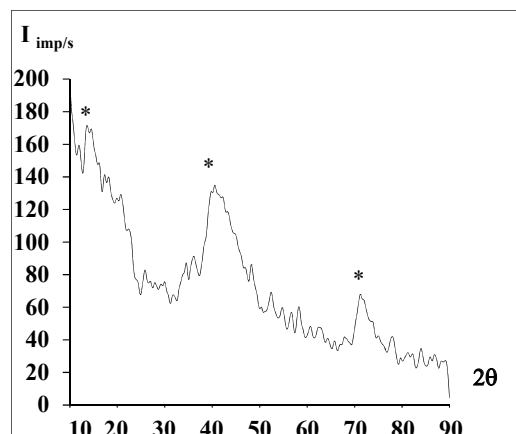


Fig. 1. Diffraction pattern of the  $\text{Ni}(\text{OH})_2$  sample obtained by low-temperature homogeneous precipitation; \* –  $\alpha\text{-Ni}(\text{OH})_2$

Fig. 2 shows SEM images of the  $\text{Ni}(\text{OH})_2$  sample obtained by low-temperature homogeneous precipitation. It is noted that the sample consists of agglomerates of spheroidal particles.

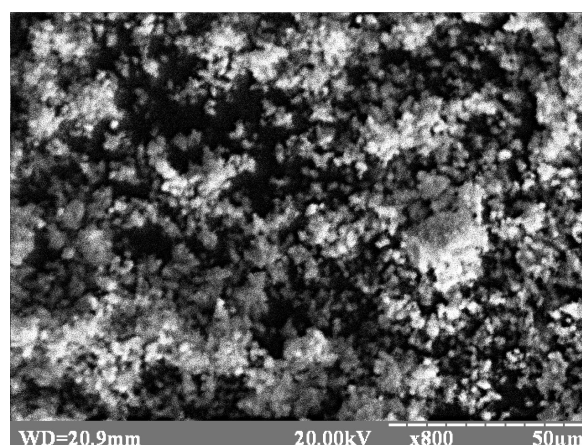


Fig. 2. SEM images of the  $\text{Ni}(\text{OH})_2$  sample obtained by low-temperature homogeneous precipitation

The cyclic voltammogram (Fig. 3) shows that the obtained sample of nickel hydroxide exhibits the characteristics of  $\alpha\text{-Ni}(\text{OH})_2$ , however, the first charge peak is not clearly pronounced.

Fig. 4 shows the specific capacities of the nickel hydroxide sample.

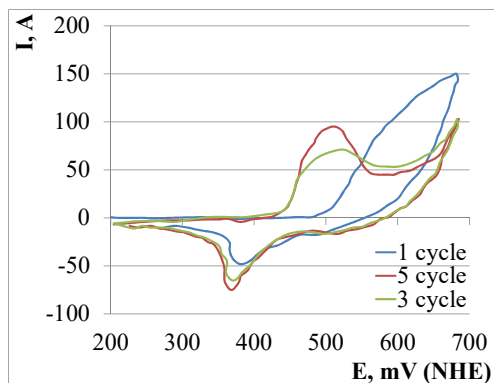


Fig. 3. Cyclic voltammogram of the Ni(OH)<sub>2</sub> sample obtained by low-temperature homogeneous precipitation

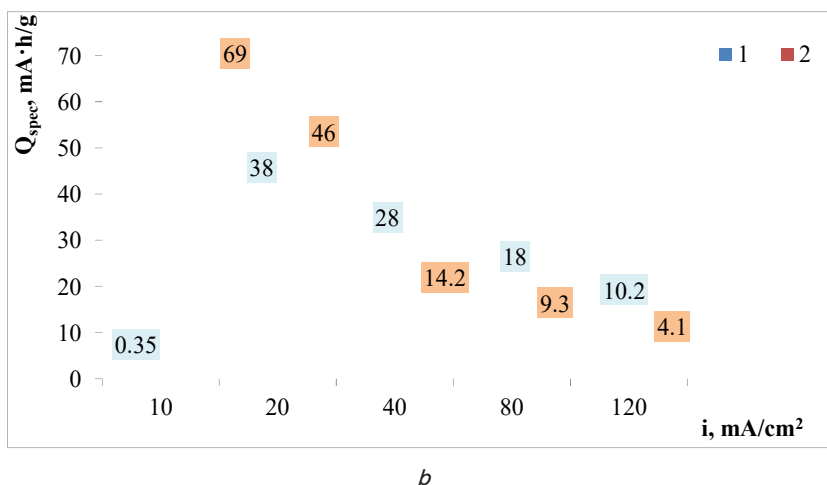
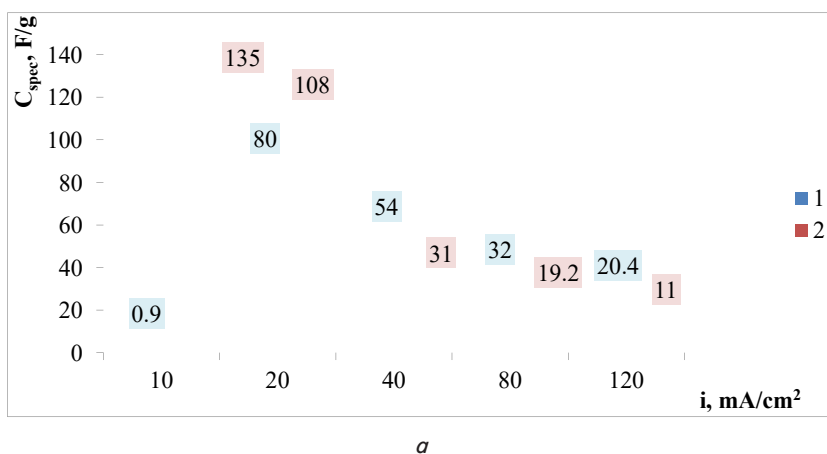


Fig. 4. Specific capacity of the Ni(OH)<sub>2</sub> sample obtained by low-temperature homogeneous precipitation; a – C<sub>sp</sub>, F/g; b – Q<sub>sp</sub>, mA·h/g; 1 – primary cycle; 2 – repeated cycle after drying for 12 hours

The data of the charge-discharge curves reveal that the capacity of the freshly made electrode (primary cycling – Fig. 4, row 1) at a low current density of 10 mA/cm<sup>2</sup> is very low, less than 1 F/g. With an increase in the cycling current density to 80 mA/cm<sup>2</sup>, the capacity increases significantly. A further increase in the cycling current density leads to a decrease in specific capacities. Repeated cycling after drying

the electrode and holding for 12 hours shows a sharp increase in specific capacities at a low current density of 10 mA/cm<sup>2</sup>. However, as the current density increases, the capacity decreases even faster than for the freshly made electrode.

### 6. Discussion of the study of the characteristics of nickel hydroxide obtained by cold template homogeneous precipitation

Visual observation shows that when the template homogeneous synthesis is carried out at room temperature, the hydroxide precipitate begins to form 30 days after the start of the synthesis. After the end of 6 months of the process, the resulting nickel hydroxide is a fine precipitate that does not have the usual hydrophilic character for Ni(OH)<sub>2</sub>. This indicates that cold template homogeneous precipitation proceeds according to a one-stage mechanism. At room temperature, the rate of hydrolysis, and, accordingly, the rate of formation of OH<sup>-</sup> ions is low. Consequently, no high supersaturation is created and the rate of crystal growth should exceed the rate of nucleation. In this case, Ni(OH)<sub>2</sub> with high crystallinity should be formed. However, the data of X-ray phase analysis (Fig. 1) show the formation of low-crystalline α-Ni(OH)<sub>2</sub>. This contradiction is explained by the influence of the water-soluble template. The scanning electron microscopy image (Fig. 2) revealed the formation of aggregates of particles of increased size. Culminar C8465 as a template, most likely, in addition to the formation of a 3D matrix, interacts with the synthesized hydroxide and exhibits the properties of a binder, causing particle aggregation, especially at high concentrations, which is consistent with the data reported in [52, 53].

The freshly made electrode shows very low capacities at a current density of 10 mA/cm<sup>2</sup>, 0.9 F/g, and 0.35 mA·h/g. With an increase in the current density to 20 mA/cm<sup>2</sup>, the specific capacity increases 88.9 times, up to 80 F/g. A further increase in the current density leads to a decrease in the specific characteristics; at a current density of 120 mA/cm<sup>2</sup>, the specific capacities are 20.4 F/g and 10.2 mA·h/g. This behavior clearly indicates the primary blocking of the particle surface by the template, which is located in the agglomerates of particles. With an increase in the current density, the agglomerates of particles disintegrate into smaller components, which leads to a sharp increase in specific capacities. These data are consistent with previously published data [19, 52]. However, with a further increase in the current density, the specific capacities decrease. This is the result of two factors. On the one hand, as shown in [19], an increase in the current density should lead to an increase in the specific capacity. But on the

other hand, a “binder-free” electrode was made for galvanostatic charge-discharge cycling. Thus, no external binder was used in the manufacture of the active mass. Probably, with an increase in the current density, with the disintegration of particle aggregates into smaller components, the binding properties of the template turned out to be insufficient. As a result, a part of the active mass may peel from the nickel-foam current collector with the loss of electrical contact. In this case, a decrease in specific characteristics indicates a stronger effect of partial peeling of the active mass.

The electrode was re-cycled after drying it at room temperature and holding for 12 hours (Fig. 4, row 2). At a current density of 10 mA/cm<sup>2</sup>, the specific capacity was 135 F/g and 69 mA·h/g. The increase in capacity compared to primary cycling was 150 and 197 times, respectively. Such a significant increase in specific characteristics is explained precisely by the decomposition of agglomerates of hydroxide particles into smaller components and an increase in the active surface. Drying the electrode in the air it leads to carbonization of the alkali in the pores of the active mass. In this case, the molar volume of sodium carbonates is greater than the molar volume of alkali. As a result, the agglomerates of nickel hydroxide particles are destroyed with an increase in the active surface and, accordingly, an increase in specific characteristics. However, an increase in the volume of the active mass can lead to partial exfoliation of the active mass with a decrease in the specific capacity. This effect was confirmed experimentally (Fig. 4): at a cycling current density of 120 mA/cm<sup>2</sup>, a specific capacity of 11 F/g was obtained, which was all 53.9 % of the specific capacity during primary cycling (20.4 F/g). Thus, it should be noted that the effect of swelling of the active mass upon drying was enhanced by the effect of insufficient binding properties of the template in the “binder-free” electrode.

In general, it should be noted that the insufficiency of the binding characteristics of the Culminal C8465 template in the “binder-free” electrode is shown, which leads to a decrease in the specific characteristics with an increase in the current density. It is necessary to carry out additional studies to clarify the amount of external binder, which has sig-

nificant properties of the binder. The previously undescribed effect of increasing the specific capacity of nickel hydroxide obtained by template synthesis upon drying the electrode after primary cycling was revealed. It is necessary to carry out additional studies of this effect with the introduction of an alkali solution during the preparation of the active mass.

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## 7. Conclusions

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1. Synthesis of nickel hydroxide has been carried out using the previously undescribed method of cold template homogeneous precipitation. A water-soluble high-molecular compound Culminal C8465 has been used as a template; the synthesis has been carried out for 6 months at a temperature of 20–35 °C. A fine powder of nickel hydroxide has been obtained.

2. Using X-ray phase analysis and scanning electron microscopy, it has been shown that during cold template homogeneous precipitation, low-crystalline  $\alpha$ -Ni(OH)<sub>2</sub> with agglomerated spherical particles is formed. The study of the electrochemical characteristics of the “binder-free” electrode with the synthesized nickel hydroxide sample has revealed high specific characteristics. The process of disintegration of agglomerates of particles into smaller ones with an increase in specific capacity has been revealed. The maximum specific capacities have been 80 F/g and 38 mA·h/g. However, the insufficiency of the binding properties of the template residues has been shown, and the conclusion has been drawn that an external binder should be introduced. The previously undescribed effect of a significant increase in the specific capacity during drying of an alkali-impregnated electrode has been revealed. The effect is due to the disintegration of particle agglomerates due to the swelling of the active mass during alkali carbonization. The maximum capacity reached has been 135 F/g and 69 mA·h/g. It has been concluded that the use of the revealed effect is promising for neutralizing the passivating effect of template residues in the composition of nickel hydroxide during template synthesis.

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