Вивчено вплив концентрації темплату Culminal C8564 на структурні, поверхневі та електрохімічні властивості зразків гідроксиду нікелю, отриманих методом темплатного гомогенного осадження. Визначено дві оптимальні концентрації темплата: 0,1 % з максимальною питомою ємністю 499 F/g та оптимальним використанням при низьких та средніх густинах струму разряду 40–80 mA/cm²; 0,4 % з максимальною питомою ємністю 525 F/g з оптимальним використанням при високих густинах струму 120 mA/cm² та вище

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Ключові слова: гідроксид нікелю, темплатний синтез, гомогенне осадження, питома ємність, суперконденсатор

Изучено влияние концентрации темплата Culminal C8564 на структурные, поверхностные и электрохимические свойства образцов гидроксида никеля, полученных методом темплатного гомогенного осаждения. Определены две оптимальные концентрации темплата: 0,1 % с максимальной удельной емкостью 499 F/g и оптимальным использованием при низких и средних плотностях тока разряда 40–80 mA/cm²; 0,4 % с максимальной удельной емкостью 525 F/g с оптимальным использованием при высоких плотностях тока 120 mA/cm² и выше

Ключевые слова: гидроксид никеля, темплатный синтез, гомогенное осаждение, удельная емкость, суперконденсатор

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1. Introduction

Supercapacitors are the most modern chemical power sources. Supercapacitors are widely used for starting electrical engines of cars, pump stations and other devices. Supercapacitors are also used as starter chemical power sources for internal combustion engines, uninterruptible power supply units for computers and other devices. Hybrid supercapacitors demonstrated the best characteristics. High charge-discharge rate is characteristic of supercapacitors. Owing to that, the electrochemical process at the Faradic electrode of supercapacitor occurs in a thin surface layer of active material. That is why, there are special requirements [1, 2] to the specific surface area, crystal structure and electrochemical activity of Faradic electrode active material. In particular, the active material must be composed of nanoand submicron-sized particles with the high specific surface area. The nickel oxide electrode with Ni(OH)₂ as an active material is used as a Faradic electrode for hybrid supercapacitors. Nickel hydroxide is used on its own [3], as nano-sized [4] or ultrafine powder [5] and as a composite with carbon nanomaterials (graphene [6], carbon nanotubes [7]).

There are various methods for the synthesis of nickel hydroxide and nickel-based layered double hydroxides. The

UDC 54.057:544.653:621.13:661.13 DOI: 10.15587/1729-4061.2017.106813

STUDY OF THE INFLUENCE OF THE TEMPLATE CONCENTRATION UNDER HOMOGENEOUS PRECEPITATION ON THE PROPERTIES OF Ni(OH)₂ FOR SUPERCAPACITORS

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hydroxide synthesis can be achieved by precipitation using titration method (addition of basic solution to solution of nickel salt) [8], precipitation at high supersaturation (addition of nickel salt to a basic solution) [9, 10]; high-temperature two-step synthesis [11], sol-gel [12]. The electrochemical methods [13, 14], including slit-diaphragm method [15, 16] are also used for synthesis.

Polymorphism is characteristic of nickel hydroxide and the two of its forms are described [17]. The β -form (chemical formula Ni(OH)₂, brucite structure) and α -form (chemical formula 3Ni(OH)₂·2H₂O, hydrotalcite structure). The β -Ni(OH)₂ possesses high cycling stability and is widely used for accumulators and supercapacitors. The α -Ni(OH)₂ possesses significantly higher electrochemical characteristics than β -Ni(OH)₂ and can be used more efficiently in hybrid supercapacitors. Thus, the development and optimization of synthesis methods for preparation of highly electrochemically active α -Ni(OH)₂ are relevant problems.

2. Literature review and problem statement

Synthesis method and its conditions directly influence the micro- and macrostructure of particles, which defines the electrochemical properties of nickel hydroxide. For effective application in supercapacitors, the nickel hydroxide has to possess specific properties [18], namely being α -Ni(OH)₂ with optimal crystallinity with submicron [5] and nanosized particles [4, 19].

The summarized reaction for nickel hydroxide synthesis can be written as follows:

$$Ni^{2+} + 2OH^{-} \rightarrow Ni(OH)_{2}.$$
 (1)

The nucleation rate is significantly higher than the rate of crystal growth. The formation mechanism of nickel hydroxide (and other precipitates of this type) consists of two stages: the first stage (very rapid) – formation of a primal amorphous particle; the second stage (slow) – crystallization (ageing) of the primal particle. As a result, the hydrophilic precipitate containing large amounts of captured mother liquor is formed. During filtration and following drying, the precipitate particles are pressed and caked, which leads to a significant increase of particle size and decrease of specific surface area. Two routes can be taken in order to prevent this phenomenon:

1) using synthesis methods with high nucleation rate and very high rate of crystal growth;

2) using special substances that prevent agglomeration of primal particles.

The first route can be realized by using the homogeneous precipitation method [20]. The essence of the method is generation of OH⁻ ions in the whole volume of the reaction solution, as a result of thermal hydrolysis of aminocompounds (urea [21, 22], hexamethylenetetramine [23]). Homogeneous precipitation can be conducted using both aqueous and mixed solvent solutions [24] or non-aqueous solutions, such as ionic liquids [25]. Usually, for the synthesis of ultrafine nickel hydroxide, the homogeneous precipitation is carried out at very high temperatures of 140–180 °C. Also, the microwave heating can be used for this purpose [26].

The second route can be realized by using surfactants [26] or template synthesis. Template synthesis is a synthesis of compounds in a template (matrix). This method is mainly used for formation of coats such as electrochromic films of Ni(OH)₂ [27, 33] or tripolyphosphate coats [28]. This leads to the formation of materials similar to polymer composites [29]. The paper [30] has demonstrated the use of water-soluble templates for the synthesis of nickel hydroxide.

However, the most promising route is the combination of two – the use of template homogeneous precipitation. It should be noted that such combined method of template homogeneous precipitation hasn't been studied in detail. Only a few water-soluble compounds were proposed for a template – PEG 6000 [31] for instance. The influence of the template concentration has not been studied at all. Therefore, the use of a water-soluble template for homogeneous precipitation and study on the influence of its concentration on the electrochemical properties of nickel hydroxide can be promising.

3. The aim and objectives of the study

The aim of the work was to optimize the composition of the solution for template homogeneous precipitation of nickel hydroxide with high specific capacity for use in supercapacitors. For the achievement of this aim, the following objectives were set:

 – synthesis of nickel hydroxide samples using the homogeneous precipitation method with different template concentrations;

 study of structural, surface and electrochemical characteristic of the prepared samples;

- comparative analysis and determination of the optimal concentration of template for preparation of highly active nickel hydroxide for use in supercapacitors.

4. Materials and methods for nickel hydroxide synthesis and characterization

4. 1. Template used for homogeneous precipitation of Ni(OH),

The template for nickel hydroxide synthesis from aqueous solution must meet several conditions. The template has to be water-soluble, high molecular compound with affinity to nickel compounds. It has been proposed to use cellulose ether Culminal C8564, which upon dissolution in water forms a 3D matrix, resulting in thickening of the solution. So, this compound can be used as a template. This compound also contains hydroxyl groups, which can react with the nickel hydroxide precipitate to form weak coordination bonds.

4.2. Nickel hydroxide synthesis method

Homogeneous precipitation by means of urea hydrolysis was used for the synthesis of Ni(OH)₂ samples. The synthesis method is described in literature [18]: a solution containing 60.9 g/l of Ni(NO₃)₂ $6H_2O$ and 229.3 g/l of urea was boiled using a water bath for 3 hours. The temperature of the reaction solution was kept at 85 °C. After the synthesis was finished, the solution was poured into a large volume of distilled water to quench the reaction. For the template homogeneous synthesis, the Culminal C8465 was used as a template. The template concentrations and sample labels are listed in Table 1.

Table 1

Template concentration and labels of nickel hydroxide samples

Template concentration, wt. % 0 0.05 0.1 0.2 0.3 0.4 0.5	Sample	0C*	0,5C	1C	2C	3C	4C	5C
	Template concentration, wt. %	0	0.05	0.1	0.2	0.3	0.4	0.5

Note: * - the reference sample was prepared without a template

After the synthesis, the prepared samples were filtered, dried at 60 °C, ground, sifted through a nickel grate, washed from soluble salts with distilled water and dried again.

4.3. Characterization of nickel hydroxide samples

The crystal structure of the samples has been studied by means of X-ray diffraction (XRD) analysis using the DRON-3 diffractometer (Russian Federation) (Co-K α radiation, range of 10–90° 2 θ , scan rate 0.1 °/s).

The shape and particle size were studied using the Scanning Electron Microscope 106 – I (SELMI, Ukraine).

The electrochemical properties of nickel hydroxide samples were studied by galvanostatic charge-discharge cycling in a special cell YSE-2 (USSR) using the digital potentiostat Ellins P-8 (Russian Federation). The working electrode was prepared by pasting a mixture of nickel hydroxide sample (82.5 % wt.), graphite (16 % wt.) and PTFE (1.5 % wt.) [32] on a nickel matrix. Electrolyte – 6M KOH. Counter electrode – nickel mesh, reference electrode – Ag/AgCl (KCl sat.). The charge-discharge cycle was conducted in the supercapacitor regime at current densities of 20, 40, 80 and 120 mA/cm² (10 cycles at each current density). The specific capacity C_{sp} (F/g) was calculated using the discharge curves.

5. Results of studying the influence of the template concentration on the characteristics of nickel hydroxide powders

The results of the XRD analysis (Fig. 1) shows that with the initial increase of the template concentration (in the sequence 0.5C-1C-1.5C-2C), the crystallinity of the samples decreases. However, with further increase of the template concentration, the crystallinity in the sample sequence 3C-4C-5C increases.

Fig. 2 shows SEM images of the samples. It is noted; that with an increase of the template concentration, the size of primal particles decreases but the degree of agglomeration increases. This is also supported by the prolonged time of the precipitate filtration with increasing template concentration.

Fig. 3 shows specific capacities of nickel hydroxide samples at different current densities of charge-discharge cycling in the supercapacitor regime.

It should be noted that control sample 0C, prepared without a template, demonstrates almost constant specific capacity of 190 F/g, with an increase of current density from 20 to 80 mA/cm², with a decrease of specific capacity being observed only after increasing the current density

to 120 mA/cm². Similar behavior is observed for samples 0.5C, 1C and 2C, prepared at low template concentrations. The capacity reaches its maximum at a current density of 80 mA/cm². It should also be noted that the specific capacity of sample 0.5C and 1C (448 F/g and 499 F/g) is significantly higher than the specific capacity of samples 0C (198 F/g). However, the specific capacity of sample 2C somewhat decreases. Samples 3C, 4C, 5C, hesized at higher template concentration showed a significantly different behavior. They show gradual (sample 3C) or rapid (sample 4C and 5C) increase in specific capacity with an increase of current density. For this group of the samples, the highest specific capacity of 525 F/g is achieved by sample 4C at a current density of 120 mA/cm².

Such specific capacity values are comparable with the values of the world's best samples [4, 5, 22].



Fig. 1. XRD patterns of nickel hydroxide samples





d

Fig. 2. SEM images of nickel hydroxide samples: a = 0,5C; b = 1C; c = 2C; d = 3C; e = 4C; f = 5C

e

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Fig. 3. Specific capacities of nickel hydroxide samples at different current densities of galvanostatic charge-discharge, F/G

6. Discussion of the results of the study on the influence of the template concentration on the characteristics of nickel hydroxide samples

The use of a template for nickel hydroxide synthesis should've decreased the particle size because of particles growing inside of the template cells, which should constrain their growth. However, for effective operation in the supercapacitor, the template should be completely removed from nickel hydroxide powder. If the template is not removed completely, it can screen nickel hydroxide particles, lowering the specific capacity. Accordingly, the discussion of the results will proceed with consideration of positive and negative influence of the template.

Influence of the Culminal C8465 template concentration on the crystallinity of the samples. The XRD patterns presented in Fig. 1 clearly demonstrated that all samples are α -Ni(OH)₂, with control sample 0C having medium crystallinity. Synthesis of nickel hydroxide with low template concentration (samples 0.5C–2C) mA/cm² leads to lower crystallinity. This is because the introduction of a template into the synthesis solution leads to the formation of a 3D matrix, with nickel hydroxide particles growing inside of its cells. The increase of the template concentration leads to reduced cell size of the matrix, which leads to a decrease of crystallinity and should decrease the particle size.

However, the further increase of the template concentration leads to an increase of crystallinity. In addition, the temperature measurements of the reaction mixture revealed the increase of temperature from 85 °C to 93 °C. The temperature increase is likely caused by a decrease of the water evaporation rate because of a thicker matrix structure. Higher temperature increases the ageing rate of nickel hydroxide, which leads to samples with higher crystallinity.

Influence of the Culminal C8465 template concentration on the morphology and size of particles. It has been found that even at minimal concentrations of a template, a significant amount of the precipitate passes through a paper filter with a pore diameter of 100 μ m. Because of that, the "Vladipor" microfiltration membranes were used in all further experiments. It has been observed that higher template concentrations lead to longer filtration times, which indicates decreasing particles sizes. This conclusion is supported by the results of Scanning Electron Microscopy (Fig. 2) that have revealed the decreasing size of primal particles with an increase of the template concentration. However, the SEM images also revealed that at the template concentration of 0.2 % and above (samples 2C, 3C, 4C, 5C), the formation of particle aggregates is observed (Fig. 2, c-f). It is likely that agglomeration and coagulation of particles occur, which can negatively impact the electrochemical performance. It can also be assumed that not the whole template had been removed from the precipitate.

Influence of the Culminal C8465 template concentration on the specific capacity of the samples. As previously discovered [18], the optimal low crystallinity and the ability of agglomerate breakdown during cycling, as well as specific surface area (particle size) have the greatest effect on the specific capacity of nickel hydroxide. The following analysis of the electrochemical activity is conducted based on these assumptions.

The data presented in Fig. 3 allow dividing the prepared nickel hydroxide samples into two groups.

The first group of samples (0C, 0.5C, 1C, 2C) was prepared without a template or at low template concentration. This group demonstrated insignificant changes in the specific capacity with an increase of the current density in the range of 20-40 - 80-120 mA/cm², reaching the highest value of specific capacity at 80 mA/ cm^2 . This indicates that these samples possess high stability and are not prone to agglomerate breakdown during cycling. It should be noted that the samples prepared at low template concentrations (0.05, 0.1 and 0.2 %) have significantly higher specific capacity, with the optimal template concentration being 0.1 % (sample 1C). It should be noted; that samples 0C, 0.5C and 0.1C have similar crystallinity (Fig. 1), but the particle size decreases in the sequence of these samples, leading to an increase of specific capacity. Crystallinity decrease for sample 2C leads to a decrease of specific capacity. For this group, the highest specific capacity value of 499 F/g is achieved by the nickel hydroxide sample prepared at the template concentration of 0.1 %.

The second group of samples (3C, 4C, 5C) had been prepared at higher template concentrations. This group demonstrates an increase of specific capacity with increasing cycling current density. For instance, 4C at a current density of 20 mA/cm² demonstrated the specific capacity of 269 F/g, and at 120 mA/cm^2 - 525 F/g (i.e. 1.95 times higher). Such behavior is characteristic of samples that are capable of agglomerate breakdown during cycling. The presence of agglomerates has been confirmed by means of Scanning Electron Microscopy (Fig. 2, d-f). It should be noted that for the second group the highest capacity is achieved by sample 4C, while samples 3C and 5C show lower capacities. This correlates with higher crystallinity of these samples. The lowest specific capacity is demonstrated by sample 5C (prepared at the highest concentration of the template), which can be explained by screening of nickel hydroxide particles by the incompletely removed template. For the second group, the maximum specific capacity of 525 F/g was achieved by the nickel hydroxide sample prepared with the template concentration of 0.4 %.

7. Conclusions

1. The structural, surface and electrochemical properties of nickel hydroxide samples prepared using homoge-

neous precipitation with different concentration of Culminal C8564 as a template have been studied. It has been found that increasing concentration of a template to 0.2 % leads to a decrease of crystallinity, while it increases with further increase of the template concentration. Increasing template concentration leads to a decrease of the particle diameter of nickel hydroxide, however, at the template concentrations higher than 0.2 %, the formation of particle agglomerates is observed. The analysis of electrochemical properties has revealed that the synthesized samples can be divided into two groups. For the first group of samples synthesized at the template concentrations below 0.2 %, there is a weak dependency of specific capacity on current density. It has been demonstrated that for this group, the maximum characteristics are defined by optimal crystallinity and decreased particle size when a template is used. The second group of the samples prepared at the template concentrations of 0.3-0.5 % demonstrated a sharp increase of specific capacity with an increase of cycling current density, which indicates the breakdown of agglomerates recorded with SEM. A sharp drop in specific capacity at a high template concentration (0.5%) has been found.

2. Upon complex analysis of the results, two optimal template concentrations of 0.1 % and 0.4 % were found. It has been demonstrated that specific capacity for the sample synthesized at the template concentration of 0.1 % was 499 F/g. This sample is recommended for cycling at medium current densities of 20-80 mA/cm². For use in high-speed supercapacitors with the current density of 120 mA/cm² and above, the template concentration of 0.4 % should be used for template homogeneous synthesis. At this template concentration, the specific capacity of 525 F/g was achieved.

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