

Гідроксид нікелю широко використовується як активна речовина суперконденсаторів. Найбільш активними є зразки  $Ni(OH)_2$  ( $\alpha+\beta$ ) шарової структури, синтезовані в щільному діафрагмовому електролізері (ЩДЕ). Однак при фільтруванні та висушуванні протікають процеси, що призводять до зниження електрохімічної активності. Вивчено вплив активуючої мікрохвильової обробки (від 0,5 до 5 хв) на структуру, морфологію поверхні, порувату структуру та електрохімічні властивості зразків гідроксиду нікелю, синтезованих в щільному діафрагмовому електролізері. Запропонована гіпотеза щодо існування «ефекту попкорну»: при короткотривалій мікрохвильовій обробці вологого зразка випромінюванням високої потужності відбудеться закипання води та мікроривування в середині зразку. Оброблені та необроблені зразки вивчені методами рентгенофазового аналізу (РФА), скануючої електронної мікроскопії та адсорбції – десорбції азоту по методу BET. Електрохімічні характеристики визначені гальваностатичним зарядно-розрядним циклуванням в режимі суперконденсатора. Існування «ефекту попкорну» було доведено збільшення товщини зразку після мікрохвильової обробки в 1,94 раза, питомої поверхні в 2,13 раза, об'єму пор в 2,66 раза та середнього радіуса пор в 1,46 раза. Показано, що збільшення часу обробки до 2–5 хв призводить до мікрохвильової сушки. Методом РФА доведено протікання процесів старіння гідроксиду нікелю при термічному сушінні та відсутність процесів старіння при мікрохвильовому сушінні та реалізації «ефекту попкорну». Як результат, утворюються рентгеноаморфні зразки. Проведено порівняльний аналіз електрохімічних характеристик оброблених та необроблених зразків  $Ni(OH)_2$ . Показано підвищення питомої ємності оброблених зразків при високій густині струму циклювання (80 и 120  $mA/cm^2$ ): при мікрохвильовому висушуванні на 10,9 %, при «ефекті попкорну» на 24–42 %. Максимальна питома ємність 231,1  $F/g$  отримана для зразка із доведеним максимальним ступенем реалізації «ефекту попкорну». Однак виявлено дезактивацію порошків гідроксиду нікелю, оброблених мікрохвильовим випромінюванням, при низьких густинах струму циклювання. Це явище пов'язано із термічною обробкою поверхні частинок при різкому нагріванні та закипанні води. Для запобігання даного негативного впливу необхідно збільшити потужність мікрохвильового випромінювання для зниження часу обробки

**Ключові слова:** гідроксид нікелю, питома ємність, суперконденсатор, мікрохвильова обробка, питома поверхня, старіння

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# “THE POPCORN EFFECT”: OBTAINING OF THE HIGHLY ACTIVE ULTRAFINE NICKEL HYDROXIDE BY MICROWAVE TREATMENT OF WET PRECIPITATE

**V. Kovalenko**

PhD, Associate Professor  
Department of Analytical Chemistry  
and Chemical  
Technologies of

Food Additives and Cosmetics\*

E-mail: vadimchem@gmail.com

PhD, Associate Professor  
Department of Technologies of  
Inorganic Substances and  
Electrochemical Manufacturing\*\*

**V. Kotok**

PhD, Associate Professor  
Department of Processes, Apparatus  
and General Chemical Technology\*

E-mail: valeriykotok@gmail.com

PhD, Associate Professor  
Department of Technologies of  
Inorganic Substances  
and Electrochemical Manufacturing\*\*

\*Ukrainian State University of  
Chemical Technology

Gagarina ave, 8, Dnipro, Ukraine, 49005

\*\*Vyatka State University

Moskovskaya str., 36, Kirov,  
Russian Federation, 610000

## 1. Introduction

Many mobile devices, especially those with motors (electric and hybrid cars, electric tools, pump stations, etc.), utilize autonomous power sources, mainly accumulators. However, for many electrical motors, the starting current exceeds the working current by 5–50 times, which lowers the effectiveness of accumulators. A modern type of chemical power sources – supercapacitors (SC) can be used to

start a motor. SC are used in uninterruptible power supplies for computers, medical equipment, critical infrastructure objects, and also as an alternative to ignition accumulators for internal combustion engines. Among all types, the hybrid supercapacitors are the most promising. Because of high charge and discharge rates of the electrode in the hybrid supercapacitor, the electrochemical reaction occurs on the surface and in a thin layer of particles of active material. Thus, there are special requirements to the active material of

the Faradic electrode, regarding specific surface area, crystal structure and electrochemical activity [1, 2].  $\text{Ni}(\text{OH})_2$  is widely used as the active material of the Faradic electrode of hybrid supercapacitors. Nickel hydroxide is employed on its own [3], as nanosized [4] or ultrafine powder [5], and as composites with nanocarbon materials [6, 7].

$\text{Ni}(\text{OH})_2$  used in supercapacitors should possess high electrochemical activity, which is governed by the following parameters [8]: type, crystallinity, particle size and morphology, specific surface area, capability of agglomerate breakdown. These parameters depend on the synthesis method and conditions. However, nickel hydroxide is also subjected to post-synthesis treatment (filtering, drying, etc.), which results in the final product. Because post-synthesis treatment also affects the characteristics of  $\text{Ni}(\text{OH})_2$ , the development of methods of post-synthesis treatment of nickel hydroxide is a relevant problem. This would allow developing directed methods for preparation of  $\text{Ni}(\text{OH})_2$ , which would include a complex “effective synthesis method + effective activating post-treatment method”.

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## 2. Literature review and problem statement

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Synthesis method and conditions directly affect the uniformity of the crystal lattice [9], microstructure [10], crystallinity [11, 12]. And in turn, these parameters significantly affect the electrochemical activity.

Firstly, the synthesis method determines the form of  $\text{Ni}(\text{OH})_2$  [13]. Nickel hydroxide is known to exist in two forms:  $\beta$ -hydroxide (chemical formula  $\text{Ni}(\text{OH})_2$ , brucite-like structure) and  $\alpha$ -hydroxide (chemical formula  $3\text{Ni}(\text{OH})_2 \cdot 2\text{H}_2\text{O}$ , hydrotalcite-like structure). However, the paper [14] describes the existence of the forms that are between  $\alpha$ - $\text{Ni}(\text{OH})_2$  and  $\beta$ - $\text{Ni}(\text{OH})_2$ .

$\alpha$ - $\text{Ni}(\text{OH})_2$  possesses higher electrochemical characteristics than  $\beta$ - $\text{Ni}(\text{OH})_2$ , however, the stability of pure  $\alpha$ -form is low, especially in concentrated alkalis at elevated temperatures. Under such conditions, the metastable  $\alpha$ -form transforms into the  $\beta$ -form [15], which results into a decrease in specific capacity.  $\alpha$ - $\text{Ni}(\text{OH})_2$  and more stable nickel-based layered double hydroxides (LDHs) can be synthesized via titration precipitation [16], homogeneous precipitation [16, 17], electrochemically in the slit-diaphragm electrolyzer [18] or cathodically deposited onto a substrate [19, 20].

$\beta$ - $\text{Ni}(\text{OH})_2$  is significantly more stable during storage and cycling, thus it is widely used in the active mass of alkaline accumulators [21] and hybrid supercapacitors [21, 22]. Synthesis of  $\beta$ - $\text{Ni}(\text{OH})_2$  can be realized chemically [21, 24], electrochemically in volume (in a slit-diaphragm electrolyzer) and on the electrode surface [23]. High-temperature synthesis is also used [25, 26].

A mixed ( $\alpha/\beta$ ) structure  $\text{Ni}(\text{OH})_2$  possesses the advantages of both forms [27, 28]. The paper [29] describes the formation of a highly active ( $\alpha+\beta$ ) form of nickel hydroxide that is different from the simple mixture of two phases. The hydroxide was synthesized in a slit-diaphragm electrolyzer (SDE) from nickel sulfate solution. The sample demonstrated high electrochemical activity, which was higher than that of  $\beta$ - $\text{Ni}(\text{OH})_2$  and  $\alpha$ - $\text{Ni}(\text{OH})_2$ . Electrochemical characteristics of ( $\alpha+\beta$ )  $\text{Ni}(\text{OH})_2$  are also improved because of high oxygen polarization upon charge [30]. High activity of layered ( $\alpha+\beta$ ) nickel hydroxide; makes electrochemical synthesis in

SDE promising for application in the complex technology “effective synthesis method + effective activating post-treatment method”.

It should be noted; that nickel hydroxide prepared via any method must be separated from the mother liquor, dried and washed from soluble salts. The nickel hydroxide that leaves the SDE has a matrix structure similar to organic-organic [31], inorganic-inorganic [32] or organic-inorganic [33, 34] composite materials, in which nickel hydroxide plays the role of the matrix with the mother liquor being a filler. Vacuum filtering or the use of press-filter causes aggregation of hydroxide particles with drying possibly resulting in ageing and thermal aggregation of particles. The paper [8] shows that the main parameter of high electrochemical activity of nickel hydroxide for application in supercapacitors is  $\alpha$  or ( $\alpha+\beta$ ) structure, optimal crystallinity, ability of agglomerates to undergo breakdown into smaller components. Upon filtering and regular thermal drying, part of these parameters (crystallinity, particle size, ability to breakdown) worsen, reducing the electrochemical activity. Various approaches for improvement of these parameters can be taken. One of the promising approaches is the development of a method that would affect water in the mother liquor and hydroxide structure while having minimal impact on  $\text{Ni}(\text{OH})_2$  itself. Microwave treatment of wet hydroxide directly after filtering is such method.

Microwave treatment is most commonly used for hydrothermal synthesis (homogeneous precipitation). In this case, the solution is heated under microwave radiation, which results in hydrolysis of ammine compounds, resulting in the basification of the solution and precipitation of hydroxide or basic salts. This can also result in oxidation as it occurs during the synthesis of  $\text{Mn}_3\text{O}_4$  [35] and  $\text{MnO}_2$  [33, 37]. Microwave-induced homogeneous precipitation is widely used for the preparation of nickel hydroxide [38]. This method was used for the synthesis of spherical  $\beta$ - $\text{Ni}(\text{OH})_2$  [39],  $\text{Ni}(\text{OH})_2$ -graphene composites [40]. Homogeneous precipitation with microwave treatment allows for the preparation of nanoparticles, such as 3D flower-like  $\text{Ni}(\text{OH})_2$  particles [41, 42] or urchin-like particles of double nickel-cobalt hydroxide [43].

Microwave treatment of the already prepared sample can have two effects. If the chosen frequency of microwave irradiation does not affect the main compound, but only water, then this would result in microwave drying [44, 45]. Or if it does affect the hydroxide, then this could result in the occurrence of the crystallochemical reaction [46, 47].

Unlike microwave-induced hydrothermal synthesis, the microwave treatment of nickel hydroxide is almost not used. This is mainly because researchers tend to ignore the processes that occur during filtering, drying, grinding. It is simply assumed that the structure and characteristics of  $\text{Ni}(\text{OH})_2$  are completely formed on the synthesis stage and all other stages are physical in nature and have no effect on the product's characteristics. However, such opinion is rather questionable, as ageing of hydroxide in the mother liquor is a well-known occurrence. However, application of microwave radiation at frequencies of water oscillation for treatment of wet, freshly-filtered nickel hydroxide could prevent ageing, increase specific surface area and increase the ability of agglomerates to undergo breakdown during cycling. At relatively high radiation power, the rate of water heating may exceed that of evaporation, resulting in boiling, buildup of internal pressure and explosion of hydroxide

particles, leading to the increased specific surface area. The authors refer to such possible scenario as “popcorn effect” in analogy to corn popping under internal pressure as a result of water boiling. If these assumptions are true, the microwave treatment can be used as an effective post-treatment for preparation of highly active nickel hydroxide.

### 3. The aim and objectives of the study

The aim of the work is to determine the possibility of improving the activity of nickel hydroxide synthesized in a slit diaphragm electrolyzer by short-time microwave irradiation of freshly filtered precipitate.

To achieve the set aim, the following objectives were formulated:

- to prepare nickel hydroxide samples in a slit diaphragm electrolyzer under optimal conditions and subject freshly-filtered samples to microwave radiation for different time periods;

- to study structural and surface characteristics of prepared samples and evaluate the possibility of achieving microwave drying and “popcorn effect” for Ni(OH)<sub>2</sub>;

- to conduct a comparative analysis of electrochemical characteristics of prepared samples and evaluate the effectiveness of microwave treatment and “popcorn effect” for obtaining highly active Ni(OH)<sub>2</sub>.

## 4. Materials and method for synthesis and study of nickel hydroxide

### 4.1. Selection of a device for microwave treatment of nickel hydroxide

Microwave treatment requires a magnetron with radiation frequency that corresponds to oscillation of water molecules. It is known that a household microwave oven utilizes the matching frequency range, and thus a household microwave oven with the frequency of 2,450 MHz and power of 1,000 W with a timer was chosen.

The “popcorn effect” can be realized at a fast heating rate and short treatment time, so that the water boils before it can evaporate. Thus, it is important to conduct treatment at the maximum possible power. It is known that microwave radiation inside the rectangular chamber is non-uniform with regions of low and high power. Thus, it has been decided to determine the region with the highest power of microwave radiation. For simplicity, only the length and height of the chamber were evaluated. The chamber was split into 2.5×2.5 cm squares. The power in each square was measured by placing a polyethylene zip bag with 5 ml of distilled water, which was heated for 5 s. After that, the water temperature was immediately measured using a thermocouple and multimeter UT-70B (Uni-T, China). In cases when water boiled during these 5 s, the measurement was repeated but for lower heating periods and was recalculated for a period of 5 s. After all cells were measured, the relative power was calculated by dividing the temperature in one cell by an average temperature in the entire cross-section. It should be noted; that the rotating glass plate was replaced by the non-rotating Teflon plate, and placement of the bags was carried out using 2.5×2.5×2.5 Teflon cubes.

### 4.2. Nickel hydroxide synthesis

The synthesis method [49] is based on electrolysis in a flow-through slit diaphragm electrolyzer (SDE). A nickel sulfate solution (Ni<sup>2+</sup> 12.7 g/L) was fed into the cathodic chamber via a peristaltic pump, and the anodic chamber was fed with NaOH (50 g/L) at the same rate of 0.2 L/h. Cathode – titanium, anode – nickel (insoluble). Electrolysis was conducted at the current density of 12 A/dm<sup>2</sup>.

The applied current leads to hydrogen evolution at the cathode and formation of hydroxyl ions, which reacts with nickel cations causing precipitation of nickel hydroxide. The precipitated was removed from the electrolyzer by the flow of catholyte. After leaving the SDE, the hydroxide precipitate was immediately vacuum filtered.

### 4.3. Treatment of the prepared hydroxide

A freshly filtered powder was separated from the filtering paper, a ~5 mm thick cake was placed into the region of the highest microwave radiation and was treated for 0.5, 1, 2 and 5 minutes. In order to separate the effect of microwave drying and “popcorn effect”, the treatment at 2 and 5 minutes was carried out in two variants:

a) exposed surface sample;

b) the sample was placed into a zip bag to prevent drying. These samples are additionally labeled with ZIP (Table 1)

Table 1

Labeling of nickel hydroxide samples

Synthesis method	Treatment duration, min	Use of ZIP bag	Label
SDE	0.5	–	MW-0.5
SDE	1	–	MW-1
SDE	2	–	MW-2
SDE	2	+	MW-2 ZIP
SDE	5	–	MW-5
SDE	5	+	MW-5 ZIP
SDE (no microwave treatment)	0	–	MW-0
Commercial sample supplied by “Bochemie”	–	–	Bochemie

All synthesized samples, microwave treated or not, were subjected to the following treatment: drying at 90 °C for a day, grinding, sifting through a 71 μm nickel mesh, washing from soluble salts and final drying.

### 4.4. Study of characteristics of nickel hydroxide samples

Crystal structure of the samples was studied by means of X-ray diffraction (XRD) analysis, using the DRON-3 diffractometer (Russia) (Co-K $\alpha$  radiation, range 10–90° 2 $\theta$ , scan rate 0.1 /s).

Sample morphology was studied using the scanning electron microscope PEMMA 120-02 (Ukraine).

Specific surface area was calculated using the BET method by low-temperature nitrogen adsorption conducted on the high-speed gas sorption analyzer Quantochrome Corp., NOVA 2200 E.

Electrochemical properties of nickel hydroxides were studied by means of galvanostatic charge-discharge cycling in a special cell YSE-2 using the digital potentiostat Ellins P-8 (Ellins, Russia). The working electrode was prepared by pasting a mixture of the nickel hydroxide sample (82.5 % wt.), graphite (16 % wt.) and PTFE (polytetrafluoroethylene) (1.5 % wt.) [50] onto the current collector. Electrolyte – 6M KOH. Counter-electrode – nickel mesh, reference electrode – Ag/AgCl (KCl sat.). Charge-discharge cycling in the supercapacitor regime was conducted at current densities of 5, 10, 20, 40, 80 and 120 mA/cm<sup>2</sup> (10 cycles at each current density). Specific capacities  $C_{sp}$  (F/g) were calculated from the discharge curves.

### 5. Results of microwave treatment of nickel hydroxide

Fig. 1 shows the distribution of the relative power of microwave radiation across the length-height cross-section. It was found; that in the 20×2.5 cm (length×height) cell, the relative power is 1/57. That is, the real power of microwave radiation is 1,000·1.57=1,570 W. This spot was chosen for treatment of all hydroxide samples.

Fig. 2 shows the thickness of the sample layers after microwave treatment. For comparison, it also shows the layer thickness of the samples prepared in the SDE that were not treated after thermal drying.

It should be noted; that the initial thickness of the samples was 4.8±0.1 mm. Data in Fig. 1 show that the layer thickness of the samples increased significantly during microwave treatment for 0.5 and 1 min (samples MW-0.5 and MW-1), which indicates swelling from the inside. The same effect is also observed for the samples placed into the zip bag before microwave treatment (samples MW-2 ZIP and MW-5 ZIP). For the samples MW-2 and MW-5, the increase in thickness did not occur, while the thickness of the sample MW-0 (not subjected to microwave radiation) has reduced significantly after thermal drying.

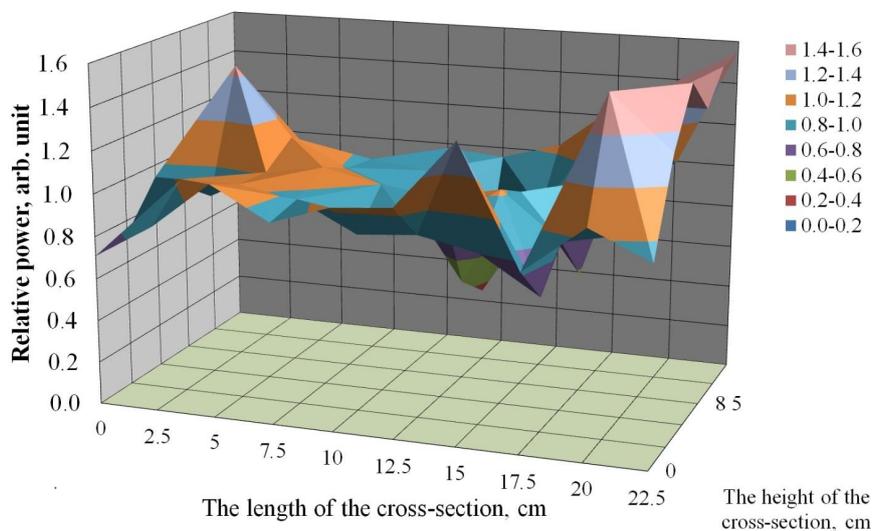


Fig. 1. Distribution of the relative power across the length-height cross-section of the microwave oven's chamber

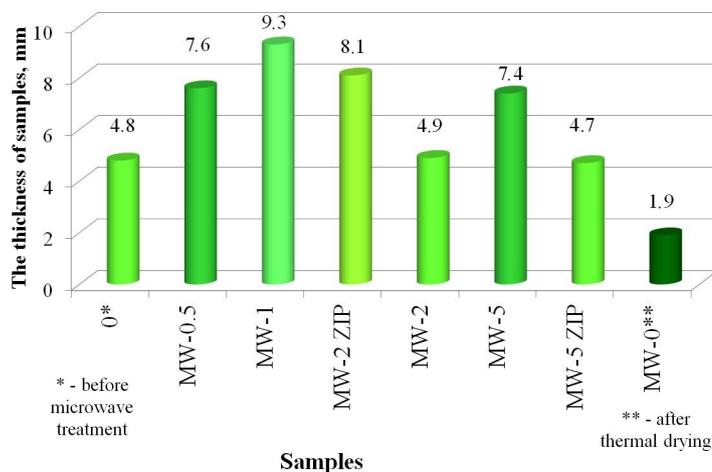


Fig. 2. Thickness of different nickel hydroxide samples

Fig. 3 shows the XRD patterns of nickel hydroxide samples prepared in the SDE after microwave treatment, and control samples (prepared in the SDE but not treated and the commercial sample).

Results of XRD analysis (Fig. 3) revealed; that the sample Bochemie is highly crystalline  $\beta$ -Ni(OH)<sub>2</sub>, and the sample MW-0 (subjected to thermal drying) – ( $\alpha$ + $\beta$ ) Ni(OH)<sub>2</sub> of average crystallinity [29]. For low durations of microwave treatment 0.5 and 1 min (MW-0.5 and MW-1) and when treated for 2 and 5 min with the use of zip bags (MW-2 ZIP and MW-5 ZIP), all samples have X-ray amorphous structure. Treatment for 2 min with exposed surface (MW-2 ZIP) does not affect crystallinity. However, during the 5 min treatment (MW-5 ZIP), the sample is not only dried, but ( $\alpha$ + $\beta$ ) layered structure with low crystallinity is formed, with predominantly  $\alpha$ -phase (peak at  $2\theta=13^\circ$ ).

Fig. 4 shows the SEM images for different Ni(OH)<sub>2</sub> samples. It should be noted; that the sample MW-0 (not subjected to microwave treatment) is composed of particle agglomerates covered with smaller hydroxide particles. Microwave treatment for 1 min (sample MW-1) results in de-agglomeration, revealing smaller components. It should be noted that for the sample MW-1, a maximum thickness increase was observed (Fig. 1). Samples treated for 5 min with exposed surface (MW-5) showed no notable changes in morphology.

Table 2 lists the specific surface area, pore volume, and diameter of different nickel samples. It should be noted; that the specific surface area of the control sample MW-0 (prepared in the SDE, but not subjected to microwave treatment) is lower than that of the control sample Bochemie. At the same time, the pore volume of the sample MW-0 is higher, and the average pore volume is significantly higher. The microwave treatment of optimal duration of 1 min (sample MW-1) resulted in the increase of all parameters: specific surface area, volume and average diameter of pores.

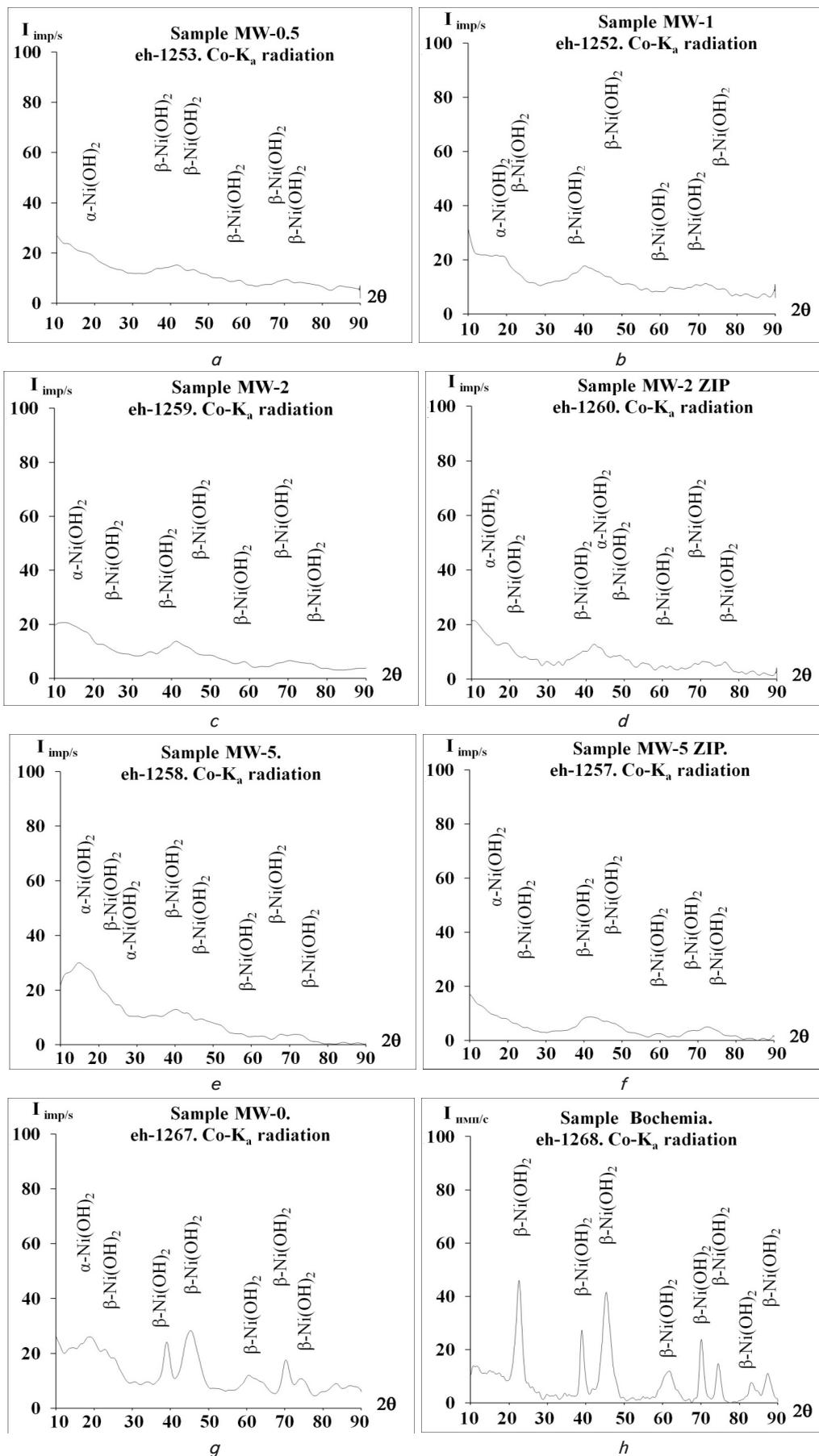


Fig. 3. XRD patterns of nickel hydroxide samples: *a* – MW-0.5, *b* – MW-1; *c* – MW-2; *d* – MW-2 ZIP; *e* – MW-5; *f* – MW-5 ZIP; *g* – MW-0; *h* – Bochemia

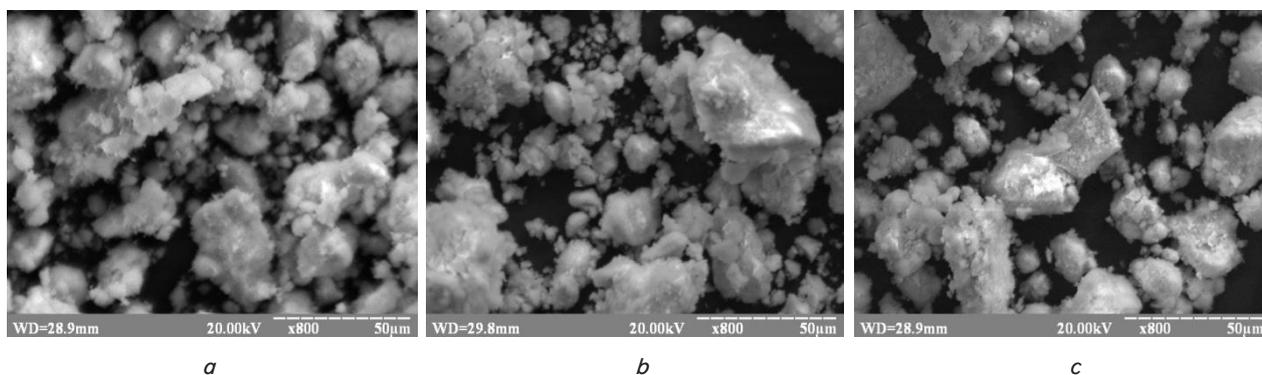


Fig. 4. SEM images of Ni(OH)<sub>2</sub> samples: *a* – MW-1, *b* – MW-0; *c* – MW-5

Table 2  
Results of the study of the porous structure of the samples by the BET method

Sample	$S_{sp}$ , m <sup>2</sup> /g	Pore volume, cm <sup>3</sup> /g	Average pore radius, Å
MW-0	57	0.18	113
MW-1	132	0.48	165
Bochemie	81	0.13	24

Fig. 4 shows the specific capacities of different Ni(OH)<sub>2</sub> samples. It should be noted; that for the commercial sample Bochemie, the specific capacity somewhat increases with the increase of current density from 20 to 40 mA/cm<sup>2</sup>, but degrades with further increase of current density. For the control sample MW-0 that was not subjected to microwave treatment, the specific capacity increases from 25 F/g at 20 mA/cm<sup>2</sup> to 165 F/g at 120 mA/cm<sup>2</sup>. For the samples treated with microwave radiation (MW-0, MW-2, and MW-2 ZIP), the dependency character of the specific capacity on the current density is preserved.

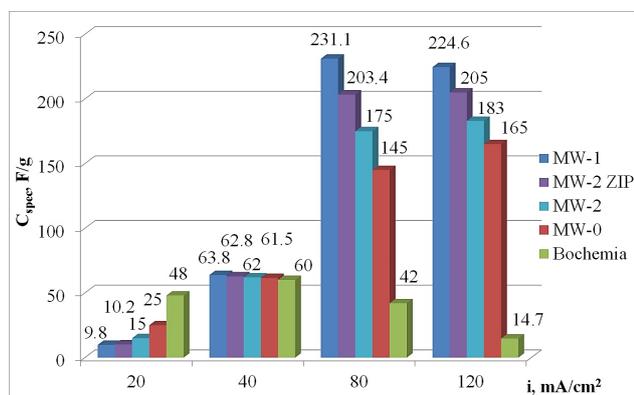


Fig. 5. Specific capacity of different nickel hydroxide samples, F/g

Two dependencies have been found. At the lowest current density (20 mA/cm<sup>2</sup>), the specific capacity increases in the series “MW-1 – MW-2 – MW-2 ZIP – MW-0 – Bochemie”. At the current density of 40 mA/cm<sup>2</sup> the capacities of all samples are similar. However, at high current densities (80 and 120 mA/cm<sup>2</sup>), the dependencies are reversed, and capacities increase in the series “Bochemie – MW-0 – MW-2 ZIP – MW-2 – MW-1”. The highest value of specific capacity is obtained for the sample MW-1 at  $i=80$  mA/cm<sup>2</sup>, 231.1 F/g.

## 6. Discussion of the results of studying the influence on microwave treatment on the characteristics of nickel hydroxide samples

*Influence of microwave treatment on the layer thickness of nickel hydroxide samples.* Measurements of the sample layer thickness before and after microwave treatment (Fig. 1) support the realization of the “popcorn effect”. After microwave treatment for 0.5 min, the layer thickness of the sample MW-0.5 increased 1.58 times, and after 1 min – 1.94 times (sample MW-1). The “popcorn” effect was realized partially, as only sample swelling was achieved. At longer treatment times (2 and 5 min), the increase in the layer thickness was only observed for the samples treated in sealed bags. The thickness increased 1.69 and 1.59 times for the samples MW-2 ZIP and MW-5 ZIP, respectively. Treatment of non-sealed samples for 2 and 5 min did not result in higher thickness. Visual observation revealed drying of the samples, especially MW-5. It can be concluded; that at longer duration, the microwave drying is the predominant effect.

*Influence of microwave treatment on the crystal structure of nickel hydroxide samples.* Results of XRD analysis (Fig. 3) revealed that microwave treatment, in case of the “popcorn effect” and microwave drying, results in decreased crystallinity of nickel hydroxide samples. Comparison of the X-ray-amorphous structure of the samples MW-0.5, MW-1, MW-2, MW-2 ZIP and MW-5 ZIP (treated with microwave radiation) and pronounced ( $\alpha+\beta$ ) structure of the sample MW-0 (prepared under the same conditions, but not subjected to microwave treatment and thermally dried) reveals the occurrence of ageing processes during regular thermal drying and their lack under conditions of the “popcorn effect” and microwave drying. Only the XRD pattern of the sample MW-5 shows the presence of ( $\alpha+\beta$ ) structure with low crystallinity. This is likely because during the longer treatment time, microwave irradiation starts to heat the hydroxide itself, especial its  $\alpha$ -Ni(OH)<sub>2</sub> part, which contains crystal water. The paper [48] describes the similar usage of microwave radiation for the transformation of  $\alpha$ -Ni(OH)<sub>2</sub> into  $\beta$ -Ni(OH)<sub>2</sub>.

*Influence of microwave treatment on the surface morphology and porosity of nickel hydroxide samples.* SEM images partially support the realization of the “popcorn effect”: particles of the sample MW-1 became swollen and start to break into their smaller components. However, the proper explosion of the particles did not occur, which is likely because of the insufficient power of microwave radiation. Treatment for 5 min (sample MW-5 with exposed surface) resulted in microwave drying, but the morphology was al-

most unaffected. The results of the BET analysis; further support the presence of the “popcorn effect”: specific surface area of the sample MW-1 increased 2.13 times, pore volume 2.66 times, and average pore diameter 1.46 times, in comparison to the untreated control sample MW-0. It should be noted that the specific surface area of the sample MW-1 is equal to  $133 \text{ m}^2/\text{g}$ , which is comparable to nanosized nickel hydroxide powders, for instance,  $173 \text{ m}^2/\text{g}$  [42]. The observed increase of surface parameters clearly shows the occurrence of microexplosions caused by local boiling of water.

*Influence of microwave treatment on the electrochemical characteristics of the samples.* As previously described [8], electrochemical activity is mainly governed by low crystallinity and ability of agglomerate breakdown into smaller components during cycling. The increase of specific surface area also improves the electrochemical activity. Analysis of data presented in Fig. 5 revealed two opposite dependencies. At high current densities; (80 and  $120 \text{ mA}/\text{cm}^2$ ), specific capacity increases in the sample series “Bochemie – MW-0 – MW-2 ZIP – MW-2 – MW-1” and is 42, 165, 183, 205 and  $231.1 \text{ F}/\text{g}$ , respectively. This directly correlates with decreasing crystallinity and increasing specific surface area upon microwave treatment. The lowest effect resulted from microwave drying, and the highest – “popcorn effect” with an optimal treatment time (1 min). The SEM images and thickness measurements revealed swelling of the sample MW-1 as a result of the “popcorn effect”. In this case, the bond strength between the aggregated particles is reduced, which facilitates agglomerate breakdown during cycling. These results in the highest capacity of the samples, in which the “popcorn effect” was realized the most. However, at the lowest cycling current density ( $20 \text{ mA}/\text{cm}^2$ ), an opposite dependency is observed: the specific capacity increases in the sample series “MW-1 – MW-2 – MW-2 ZIP – MW-0 – Bochemie”. The samples, in which the “popcorn effect” was realized the most, showed the lowest capacity. This is likely the result of surface heating of particles, caused by water boiling, which possibly resulted in the formation of less active  $\beta\text{-Ni}(\text{OH})_2$  on the surface of the amorphous phase. At the current density of  $40 \text{ mA}/\text{cm}^2$ , capacities of all samples are very close, as both the “popcorn effect” and thermal treatment of the surface balance each other. This leads to the assumption that higher power of microwave radiation and lower treatment time would result in the lower effect of thermal influence.

*Summary of the results obtained on microwave treatment of wet precipitate.* Analysis of the obtained results on microwave treatment of the freshly filtered nickel hydroxide precipitate has proven the realization of the “popcorn effect”. Under conditions at which the “popcorn effect” if realized, it is possible to obtain more electrochemically active nickel hydroxide, not from activation, but through prevention of passivation as a result of re-crystallization. It should be noted, that full realization of the “popcorn effect” was not achieved, and a side-effect of partial surface passivation of nickel hydroxide was observed. Industrial-scale implementation would require the determination of optimal conditions

for realization of the “popcorn effect”, which is the aim for future studies. However, the current data allow outlining possible applications. Microwave treatment of wet precipitates under conditions at which the “popcorn effect” is realized (high power microwave radiation and low treatment time) allows preparing ultrafine powders with high specific surface area. However, the treated materials should be stable under employed frequencies of microwave treatment. The “popcorn effect” can also be used for destruction of cellular water-containing materials. The microwave treatment would simplify further extraction of necessary compounds, which would find application for the opening of some materials or as pretreatment for analysis.

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## 7. Conclusions

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1. The influence of microwave treatment of different times (from 0.5 to 5 min) on the structure, surface morphology and porous structure, and also on the electrochemical properties of nickel hydroxide samples prepared in a slit diaphragm electrolyzer has been studied. The results of BET and layer thickness measurements have confirmed the existence of the “popcorn effect” (internal explosion of nickel hydroxide under short-time high-power microwave irradiation). After 1 min of treatment, the thickness of the sample layer increased 1.94 times, specific surface 2.13 times, pore volume 2.66 times and average pore diameter 1.46 times. However, it should be noted; that the full realization of the “popcorn effect” has not been achieved, which only resulted in the swelling of the nickel hydroxide sample, as revealed by the SEM results. It has been discovered; that increasing treatment duration to 2–5 min results in microwave drying. XRD results revealed the occurrence of ageing (crystallization) processes of nickel hydroxide during thermal drying. The occurrence of ageing processes during microwave drying and realization of the “popcorn effect” has not been observed.

2. A comparative analysis of electrochemical characteristics of  $\text{Ni}(\text{OH})_2$  samples, subjected to microwave treatment of different duration was conducted. An increase of specific capacity at high current densities ( $80$  and  $120 \text{ mA}/\text{cm}^2$ ) for the treated samples was observed. Two minutes of microwave drying resulted in an increase of specific capacity by 10.9 %. A stronger influence of the “popcorn effect” was observed: increase in specific capacity by 24.2 % and 42 % from 2 min and 1 min of treatment, respectively. The maximum capacity of  $231.1 \text{ F}/\text{g}$  has been achieved for the sample treated by microwave radiation for 1 min. This sample also demonstrated the highest increase in the layer thickness and the highest realization of the “popcorn effect”. However, microwave treatment resulted in lower capacities at low cycling current density. This is related to the thermal treatment of the particle surface, caused by rapid boiling of water. A magnetron of a higher power is required for avoiding this negative effect.

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