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ELECTROCHEMICAL SYNTHESIS OF NICKEL HYDROXIDE FROM NITRATE SALT IN THE SLIT DIAPHRAGM ELECTROLYZER FOR POWER SOURCES APPLICATION

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ABSTRACT

$Ni(OH)_2$ is widely used as active material for Faradic electrode in super-capacitors. In this study, it was found that synthesis of nickel hydroxide synthesis in slit-diaphragm electrolyzer from nickel nitrate solution as catholyte is possible. It was discovered that synthesis only possible at low current densities (4-6 A/dm^2) due to overheating of catholyte. Analysis of synthesized samples revealed that have low crystallinity layered (α+β) $Ni(OH)_2$ structure, with increased content of α-$Ni(OH)_2$. A formation of phase that is intermediate between α-$Ni(OH)_2$ and β-$Ni(OH)_2$ was discovered. Cyclic voltammetry studies revealed high electrochemical activity and cyclic stability of synthesized samples. It was noted, that further studies are needed to refine the proposed method for industrial applications.

Keywords: nickel hydroxide, (α+β) $Ni(OH)_2$, nitrate solution, slit diaphragm electrolyzer, neither α-$Ni(OH)_2$ nor β-$Ni(OH)_2$, voltammogram.

1. INTRODUCTION

$Ni(OH)_2$ is an electrochemically active compound [1] and is widely used in various electrochemical devices. Nickel hydroxide and nickel-based layered double hydroxides are used as active materials for nickel oxide electrode in alkaline Ni-Cd, Ni-Fe and Ni-MeH accumulators [2, 3]. Nickel hydroxide is also used in cathodes of lithium batteries [4].

$Ni(OH)_2$ is widely used as active material for Faradic electrode in supercapacitors. It used individually, [5], as nanosized [6] or ultrafine powder [7], and as a composite with nanocarbon materials (graphene oxide [8], carbon nanotubes [9]). For thin layer capacitors, nickel hydroxide film is formed on the surface of the conductive substrate [10].

Due to significant color change during redox transformations, (a thin film of $Ni(OH)_2$ is transparent, and NiOOH is dark-brown), nickel hydroxide is used in electrochemical devices [11, 12].

Electrochemical activity of nickel hydroxide enables its use for electrochemical oxidation of organic compounds [13, 14], sensors as well [15, 16].

Two forms of nickel hydroxides have been described in [17]. β-form (chemical formula $Ni(OH)_2$, brucite crystal structure) and α-form (chemical formula $3Ni(OH)_2 \cdot 2H_2O$, hydrotalcite crystal structure). The study [18] also describes a series of intermediate nickel hydroxide structure. It worth noting, that the synthesis method determines the form of nickel hydroxide [40], and its characteristics [35, 37, 39, 45]. β-$Ni(OH)_2$ possess high stability and be synthesized by reverse titration [19], ammine complex decomposition [20] or high-temperature two-stage synthesis [21]. α-$Ni(OH)_2$ can be synthesized using many methods, for instance, homogeneous precipitation [22], including from mixed solvents [23] or under vacuum [24], and microwave synthesis [25]. A thin film of nickel α-$Ni(OH)_2$ can be prepared by cathodic deposition, including template synthesis [26]. This form is more electrochemically active but is less stable than β-$Ni(OH)_2$. To stabilize α-form, different additives are used during the synthesis stage, namely cations of other metals [27, 47, 48, 51, 52]. Another effective way is the formation of layered double hydroxides (LDH), in which part of nickel cations is substituted with tri- or a tetravalent cation, with an excess of positive charge, compensate by addition anions: Al^{3+} and CO_3^{2-} [28, 29], Ti^{4+} and CO_3^{2-} [30].

α-$Ni(OH)_2$ is more electrochemically active than β-$Ni(OH)_2$. However, its stability is significantly lower. As such the most promising material for different application is a material containing both α-$Ni(OH)_2$ and β-$Ni(OH)_2$. Paper [31] describes the synthesis of pure β-$Ni(OH)_2$, coated with Al-stabilized α-$Ni(OH)_2$. This resulted in a material with increased capacity. Even more promising is the synthesis of mixed α/β-phase, which should possess even higher electrochemical materials. Such mixed phases were synthesized using additives of Al^{3+} [32], Ca^{2+} and PO_4^{3-} [33]. But these phases contain additional inert ions.

Previously [34], nickel hydroxide samples with high electrochemical activity were synthesized in slit-diaphragm electrolyzer (SDE). XRD analysis reveals the presence of α-$Ni(OH)_2$ and β-$Ni(OH)_2$ and the hypothesis of its layered structure were formulated. In paper [42], this hypothesis was proven by contradiction. Physical modeling was conducted by preparing physical mixtures with different content of α-$Ni(OH)_2$ and β-$Ni(OH)_2$. Study of mixture samples revealed that samples synthesized in SDE are not just a mixture of α-$Ni(OH)_2$ and β-$Ni(OH)_2$, but have layered (α+β) structure.

It should be mentioned, that hydroxide synthesized in SDE has a matrix structure, similar to organic [41] and inorganic [49] composite materials. This corresponds to the formation mechanism of nickel hydroxide, which is described by Wasserman [38]. As described in [43, 44], nickel hydroxide plays the role of a matrix with mother liquor being a filler. During synthesis, primal particles move inside slit of SDE and age in the field of Joule heat, which is resulted from electrical current flowing between anode and cathode. Such a combination of particle growth and partial crystallization from aging is the reason behind the formation of unique (α+β) layered structure. A significant role in the formation and stabilization of this structure is also played by anions. Influence of anions on properties of LDH is studied rather well [47]. In particular, paper [48] describes the positive effect of nitrate-ion on electrochemical characteristics of Ni-Al LDH. One the way for improving the electrochemical activity of (α+β) layered structure is by increasing content of α-form. It is known, that synthesis from nickel nitrate primarily results in α-$Ni(OH)_2$.

The aim of the research is to determine the possibility of electrochemical synthesis of nickel (II) hydroxide in slit-diaphragm electrolyzer from nickel nitrate solution and to study its properties.

2. MATERIALS AND METHODS

2.1 Synthesis of nickel hydroxide samples

Nickel hydroxide samples were synthesized using slit-diaphragm electrolyzer (SDE), which is composed of cathodic and anodic half-elements, low-permeability diaphragm Doramik (to allow income of carbonate ions [46]), and two rubber gaskets, forming near-electrode space (Figure-1). Anodic space of SDE was fed with 50 g/L NaOH solution, cathodic space –$Ni(NO_3)_2$ solution, Ni^{2+} 12.7 g/L. Solutions were fed using peristaltic pumps NP-1M. The synthesis was conducted in a galvanostatic regime at current densities of 4 - 12 A/dm^2. Formation if OH^- occurs at cathode according to the following equations:

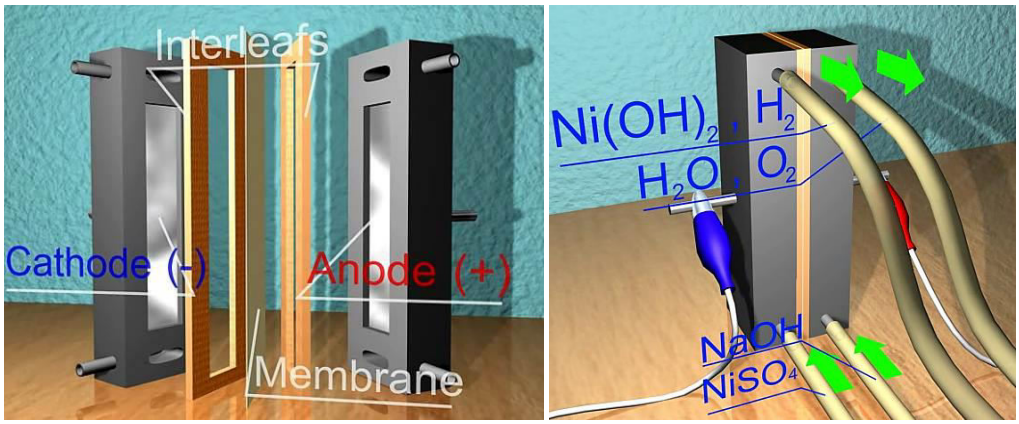
$$2NO_3^- + 6H_2O + 10e^- = NO_2^- + 12OH^- \quad (1)$$

$$NO_3^- + H_2O + 2e^- = NO_2^- + 2OH^- \quad (2)$$

$$H_2O + 2e^- = H_2 + 2OH^- \quad (3)$$

Catholically generated OH^- ions then react with Ni^{2+} ion in volume, resulting in the formation of hydroxide. The formed precipitate is partially deposited onto the cathode, and the rest is washed out of apparatus and was immediately separated from the mother liquor by vacuum filtration. Acidification of electrolyte occurred at the anode.

All synthesized samples were subjected to the following treatment:
first drying (80 ºC) → grinding → sifting through 71 µm mesh → washing from soluble salts in distilled water for a day → filtering and second drying.



a) b)

Figure-1. Slit-diaphragm electrolyzer: a) components, b) assembled.

2.1 Analysis of prepared samples

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

Electrochemical characteristics of nickel hydroxide samples were studied by means of cyclic voltammetry in special cell YSE-2, using digital potentiostat Elins P-8 (Elins, Russia). The working electrode was prepared by pasting a mixture of nickel hydroxide (81 % wt.), graphite (16 % wt.) and PTFE (polytetrafluoroethylene) (3 % wt.) [36] onto the current collector. Current collector was prepared by welding Ni mesh onto a Ni plate. Electrolyte - 6M KOH. Counter-electrode -nickel mesh, reference electrode - Ag/AgCl (KCl sat.). Cyclic voltammograms were recorded in the potential widow of 0-500 mV (NHE) at a scan rate of 1 mV/s.

3. RESULTS AND DISCUSSIONS

It is important to mention that when the synthesis of nickel hydroxide in SDE from nitrate solution was

attempted at current densities of 8-12 A/dm^2 (optimal for synthesis from sulfate solution [34, 42-44, 46]), significant overheating of catholyte, and even boiling was observed. This can be explained by the significant difference in ΔH of reactions (1-2) in comparison to ΔH of reaction (3). As such, synthesis of nickel hydroxide from nitrate solution in SDE was only conducted at current densities of 4 and 6 A/dm^2. A significant amount of nickel hydroxide was deposited onto cathode and remained inside SDE, which is a significant disadvantage.

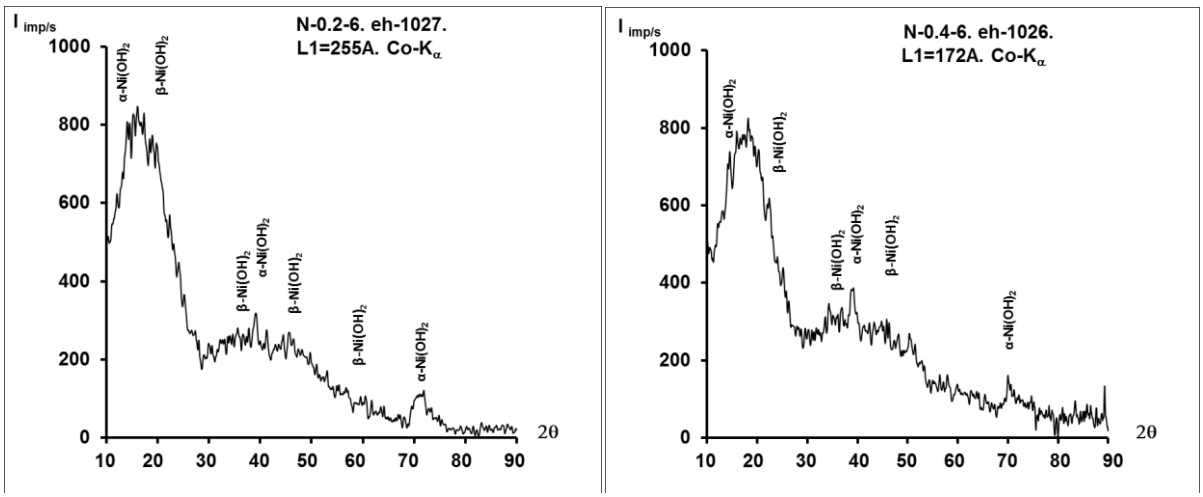
Table-1 lists the labelling of prepared nickel hydroxide samples.

Table-1. Labelling of nickel hydroxide samples.

Label	**N-0.2-4-E**	**N-0.2-6**	**N-0.4-6**	**N-0.4-6-E**
Current density, A/dm^2	**4**	**6**	**6**	**6**
Catholyte flow rate, L/h	0.2	0.2	0.4	0.4
Note	The sample removed from the electrode	Sample filtered off from catholyte	Sample filtered off from catholyte	The sample removed from the electrode

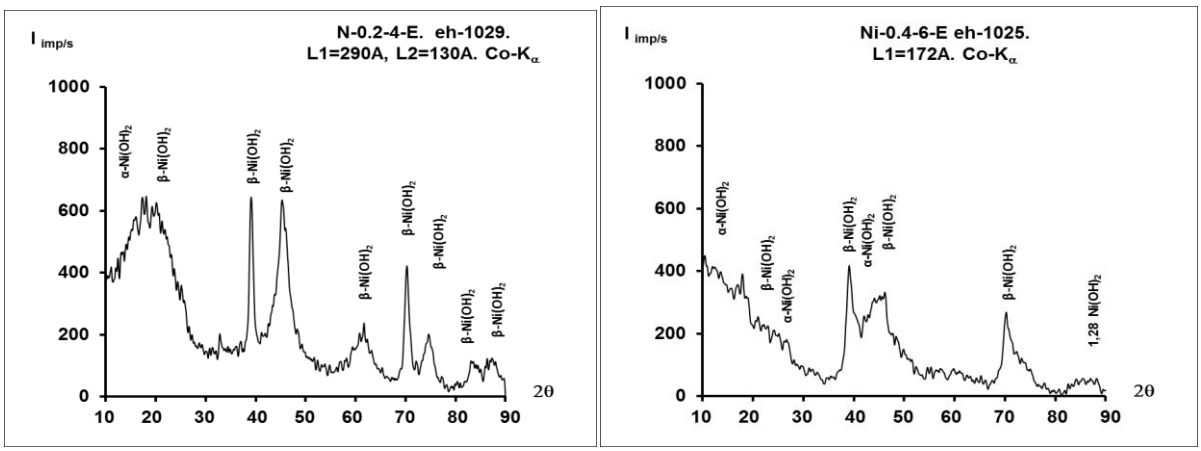
Sample structure.

XRD patterns of sample N-0.2-6 (Figure-2a) and N-0.4-6 (Figure-2b) show that samples are low crystallinity (α+β) $Ni(OH)_2$, with increased content of α-$Ni(OH)_2$. It is worth to note, that XRD pattern shows peaks of α-$Ni(OH)_2$ (13°) and β-$Ni(OH)_2$ (21°), and phase peaks at 15-17°. This indicates the formation of an intermediate phase, with spacing c that is intermediate between α and β. This phase contains less crystal water than α-$Ni(OH)_2$, with chemical formula $3Ni(OH)_2 \cdot 2H_2O$. Sample N-0.2-6 and N-0.4-6 contain some amount of X-ray amorphous β-$Ni(OH)_2$, which is evidenced by broadening of the first peak. Nickel hydroxide samples removed from the electrode, N-0.2-6-E (Figure-3 a) and N-0.4-6-E (Figure-3b), are also (α+β) $Ni(OH)_2$. But the content of β-$Ni(OH)_2$ is higher(appearance of pronounced β phase peaks), and crystallinity is also higher. For sample N-0.2-6-E high peak of an intermediate phase between α and β is observed. XRD pattern of sample N-0.2-6-E (Figure-3a) shows a pronounced peak of β-$Ni(OH)_2$ with high crystallinity. This is likely caused by additional heating of $Ni(OH)_2$ that remained in apparatus, which resulted in accelerated aging [53]. At the same time, on XRD pattern of sample N-0.4-6-E, the peak of the intermediate phase is not the most pronounced, and the peak of α-$Ni(OH)_2$ is more intense. β-$Ni(OH)_2$ peaks are less pronounced. All of this is explained by the lower aging rate for hydroxide on the electrode, due to lower electrolyte temperature because of higher catholyte flow rate.



a) b)

Figure-2. XRD patterns of samples: a) N-0.2-6; b) N-0.4-6.



a) b)

Figure-3. XRD patterns of samples: a) N-0.2-4-E; b) N-0.4-6-E.

Electrochemical properties of samples.

Cyclic voltammograms of all samples show character similar to layered (α+β) $Ni(OH)_2$. A first charge peak is observed on cyclic curves. However, for samples prepared at a lower flow rate (N-0.2-4-E and N-0.2-6), first charging peak is less defined (Figure-4 a, b), while first cathodic peak is well-defined and is characterized by the significant current. This indicated that during the first charge that nickel hydroxide is primarily oxidized

For samples prepared at a higher flow rate (N-0.4-6 (Figure-3 c) and N-0.4-6-E (Figure-3 d)), first oxidation peak is more pronounced, which indicates a significant contribution of the electrochemical process into initial oxidation, which is characteristics of α-$Ni(OH)_2$. This data correlates with XRD patterns that indicated a predominance of X-ray amorphous α-nickel hydroxide. On second and subsequent cycles stability of cathodic peaks is observed for all samples, which indicates breaking in of hydroxide and high polarization of oxygen evolution. Obtained data indicates high electrochemical activity of obtained layered (α+β) $Ni(OH)_2$ samples with increased content of α-$Ni(OH)_2$.

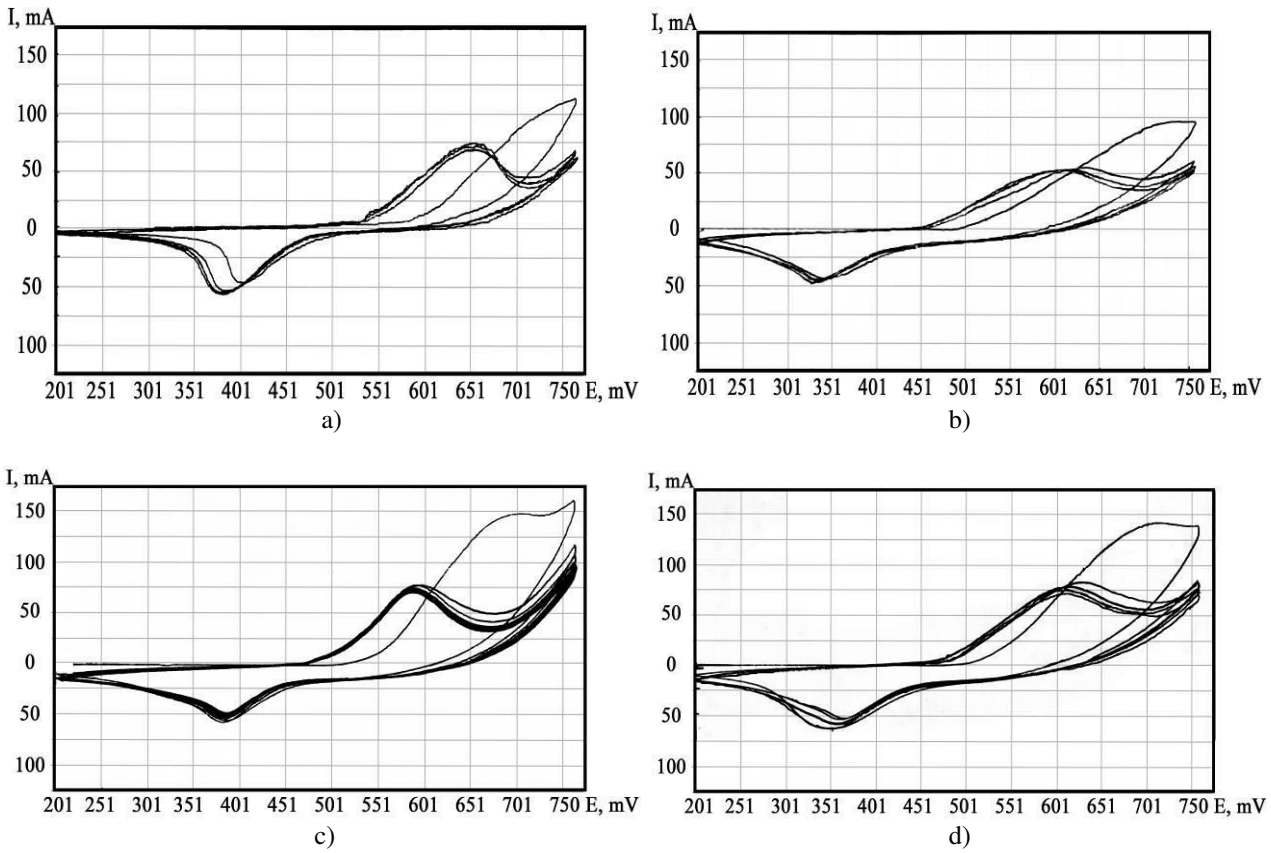


Figure-4. Voltamperogram of samples: a) N-0.2-4-E; b) N-0.2-6; c) N-0.4-6; d) N-0.4-6-E.

4. CONCLUSIONS

a) Samples of nickel hydroxide were prepared from nitrate solution in the slit-diaphragm electrolyzer. Characteristics of prepared samples have been studied.

b) It was found that synthesis of $Ni(OH)_2$ of in SDE from nickel nitrate solution (Ni^2+ 12.7 g/L) is only possible at low current densities (4-6 A/dm^2) due to electrolyte overheating. It is noted, that significant amount of hydroxide remain inside of electrolyzer, which is a disadvantage.

c) Results of XRD analysis have revealed that prepared samples have layered (α+β) structure, with increased content of low crystallinity α-structure. Amount of β-phase and crystallinity increases with a lower flow rate and increased current density. Formation of structure that is intermediate between α and β form was found for the first time. Cyclic voltammetry studies revealed high electrochemical activity and cyclic stability of synthesized samples.

d) Samples synthesized from nitrate solution in slit-diaphragm electrolyzer show promise for application in alkaline accumulators. However, underused synthesis conditions, (nickel concentration, flow rate, and current density), the process cannot be upscaled due to thermal instability and difficulties with maintaining continuous production. Further study is needed for solving these issues.

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