

Electrochemical devices based on nickel hydroxide electrodes are used in different areas. The main ones are chemical current sources, variable transparency "smart" windows, devices for carrying out electrocatalytic reactions, sensors for determining various substances. In this regard, methods of nickel hydroxide synthesis are of great interest, especially those that allow forming nickel hydroxide directly on the surface of electrodes. One of these methods is electrochemical deposition with cathodic current polarization.

The available information on nickel hydroxide synthesis from nickel solutions was considered. It was shown that the available data mainly covered information on dilute solutions from 0.01 to 0.25 mol/L Ni(NO₃)₂. In addition, no comparison was found in the literature for the efficiency of the cathodic formation of Ni(OH)₂ at different concentrations of nickel nitrate.

To eliminate the lack of information, the dependence of the current efficiency on the concentration of nickel nitrate in the electrodeposition solution was determined at a constant cathode current density of 0.625 mA/cm². The resulting dependence decreased nonlinearly with increasing concentration. The nickel hydroxide deposit formed in this case had an X-ray amorphous structure, and it depended little on the Ni(NO₃)₂ concentration. In addition, the current efficiency reached zero at concentrations of 1.5 mol/L Ni(NO₃)₂ and higher. However, with polyvinyl alcohol in the solution and at Ni(NO₃)₂ concentrations of 1.5 and 2 mol/L, electrochemically and electrochromically active Ni(OH)₂ films were deposited. The current efficiency calculated indirectly for 1.5 and 2 mol/L Ni(NO₃)₂ solutions was 3.2 and 2.3 %, respectively. Thus, it was concluded that polyvinyl alcohol affected the mechanism of nickel hydroxide electrodeposition from aqueous solutions of nickel nitrate

Keywords: electrochromism, electrodeposition, nickel hydroxide, polyvinyl alcohol, nickel nitrate, current efficiency

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1. Introduction

Nickel hydroxide (Ni(OH)₂) has been widely used in various electrochemical systems due to its unique properties. Since, as a result of an electrochemical reaction, Ni(OH)₂ can repeatedly and reversibly transform into NiOOH with high efficiency, nickel hydroxide has found application in the

production of batteries of Ni-MH [1], Ni-Fe [2], Ni-Cd [3], Ni-Zn [4] and Ni-H₂ systems [5].

It is possible to obtain Ni(OH)₂ powders with a high specific surface [5], so this material is used in the composition of the nickel oxide electrode of asymmetric supercapacitors [6, 7]. In this case, the capacitance given off at high cycling rates of supercapacitors based on Ni(OH)₂ is the sum

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EFFICIENCY DEFINITION OF THE DEPOSITION PROCESS OF ELECTROCHROMIC Ni(OH)₂-PVA FILMS FORMED ON A METAL SUBSTRATE FROM CONCENTRATED SOLUTIONS

Valerii Kotok

Corresponding author

PhD, Associate Professor

Department of Processes, Apparatus and General Chemical Technology*

E-mail: valeriykotok@gmail.com

Vadym Kovalenko

PhD, Associate Professor

Department of Analytical Chemistry and Chemical Technology of Food Additives and Cosmetics*

Rovil Nafeev

PhD, Associate Professor

Department of Physics**

Volodymyr Verbitskiy

Doctor of Pedagogical Sciences, Professor

Department of Medical, Biological and Valeological Basics of Life and Health Protection

National Pedagogical Dragomanov University

Pyrohova str., Kyiv, 9, Ukraine, 01601

Director

National Ecological and Naturalistic Center for Student Youth

Vyshhorodska str., 19, Kyiv, Ukraine, 04074

Olena Melnyk

PhD, Associate Professor, Senior Researcher

Research Coordination Office***

Iryna Plaksiienko

PhD, Associate Professor

Department of Ecology, Sustainable Nature Management and Environmental Protection

Poltava State Agrarian University

Skovorody str., 1/3, Poltava, Ukraine, 36003

Igor Kovalenko

Doctor of Biological Sciences, Professor, Dean of Faculty

Department of Ecology and Botany***

Viktoriiia Stoliarenko

PhD, Associate Professor

Department Chemistry and Methods of its Teaching

Kyryvi Rih State Pedagogical University

Gagarina ave., 54, Kryvyi Rih, Ukraine, 50086

Valerii Plaksiienko

Doctor of Economic Sciences, Professor

Department of Accounting and Economic Control

Poltava State Agrarian University

Skovorody str., 1/3, Poltava, Ukraine, 36003

Iryna Zamrii

PhD, Associate Professor

Department of Higher Mathematics, Mathematical Modeling and Physics**

*Ukrainian State University of Chemical Technology

Gagarina ave., 8, Dnipro, Ukraine, 49005

**State University of Telecommunications

Solomenska str., 7, Kyiv, Ukraine, 03110

***Sumy National Agrarian University

Herasyma Kondratieva str., 160, Sumy, Ukraine, 40000

of the capacitance of the reversible electrochemical reaction of the $\text{Ni(OH)}_2/\text{NiOOH}$ pair and the charge capacitance of the double electric layer. The latter is determined by the specific surface area of nickel (II) hydroxide.

In addition, hydroxide-oxide nickel films formed by different methods [8] are widely used as electrochromic coatings [9]. These coatings can reversibly change their optical characteristics since nickel hydroxide in thin layers is transparent and transforms into a dark brown oxyhydroxide NiOOH upon oxidation with an electric current [10].

Nickel oxide electrode is also positioned as an element of electrochemical cells for oxidation from organic wastewater contamination [11]. This application is due to the fact that, during anodic polarization, a constantly renewable film of an active electrocatalyst for oxidation is formed. In [12], modified nickel hydroxide is used to oxidize urea in a fuel cell, and [13] shows an example of the oxidation of some carbohydrates as alternative fuels.

Nickel hydroxide in the composition of electrodes has also found application in sensor systems [14], including for determining antibodies [15].

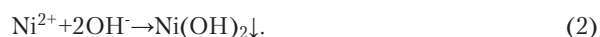
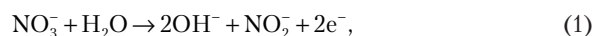
The analysis of the areas of its use is broad and not limited to those listed. As an example of a specific application of nickel hydroxide, one can name the use of the latter to increase the dielectric constant of vulcanized silicone rubber [16].

Considering all the variety of forms and purposes of application, it can be argued that the study of Ni(OH)_2 production methods is an important research task.

2. Literature review and problem statement

Nickel hydroxide is deposited by various methods, among which the electrochemical one stands out [17]. As a result of the electrochemical reaction, OH^- ions are released at the cathode, and they interact with Ni^{2+} ions, resulting in the formation of a hardly soluble deposit of nickel (II) hydroxide. The initial components of the solution, which give hydroxyl ions, are either water molecules or a combination of water molecules with a nitrate ion. Less commonly, the method of destruction of a previously prepared ammonia complex of nickel through acidification of the solution at the anode is used. However, it is difficult to work with the ammonia complex of nickel, since as a result of the evaporation of ammonia, it is easily destroyed and the necessary deposit is formed not only on the anode but also on the walls of containers. Therefore, the most convenient is the cathodic production of hydroxide deposits.

It should be noted that when water decomposes in aqueous solutions, hydrogen is simultaneously formed. The latter can lead to exfoliation of the deposit on the cathode or the formation of uncovered places because some of the hydrogen bubbles remain on the electrode and block its surface. Therefore, the most rational is the use of nitrate aqueous solutions, since there are no gases during cathodic evolution:



The advantage of the electrochemical method is the simplicity of its implementation, the absence of the need for powerful energy sources, ease of control over the properties of the resulting deposits, as well as excellent automation [18].

It should be noted that electrochemical deposition from aqueous solutions of nickel salts is often used to form electrodes. Thus, in [19], Ni(OH)_2 films were formed on nickel from solutions of 0.1 mol/L nickel chloride, nitrate, and sulfate to study the parameters of the resulting structures. In [20], researchers obtained mesoporous films of Ni(OH)_2 from a 0.02 mol/L $\text{Ni(NO}_3)_2$ solution with the addition of surfactants and water-soluble polymers. The formation of films on nickel foil for supercapacitors was studied in [21]. In this case, a 0.08 mol/L $\text{Ni(NO}_3)_2$ solution was used.

To study the efficiency of the deposition process on different substrates (nickel and platinum), as well as the effect of cathode current density, 0.1 mol/L $\text{Ni(NO}_3)_2$ was used [22]. At the same time, $\text{Ni(NO}_3)_2$ solutions in the range of 0.25–1 mol/L were used in [23] to determine the composition and structural parameters. At the same time, the authors did not consider the efficiency of the electrodeposition process.

In [24], electrochromic films of nickel hydroxide from a 0.01 mol/L $\text{Ni(NO}_3)_2$ solution were studied. For use in solar cells, other researchers formed films of nickel hydroxide in 0.2 mol/L $\text{Ni(NO}_3)_2$ dissolved in pure dimethyl sulfoxide and mixtures of dimethyl sulfoxide with water.

It is worth noting that the listed works used relatively dilute concentrations of nickel salts. Although it would be technologically more correct to form films from more concentrated solutions since this would reduce the relative change in the concentration of the ion source during the operation of the solutions. As a consequence, a decrease in concentration would have less effect on changes in film structure upon prolonged deposition of Ni(OH)_2 . In addition, more time would be needed between adjustments or complete change of electrolyte.

During the search, only a few papers were found in which the deposition was carried out from relatively concentrated electrolytes.

A group of researchers in [25] formed nickel hydroxide films with soluble polymers for supercapacitor electrodes, as well as for electrochromic devices [26]. Also in [27], nickel hydroxide was deposited from 1.8 mol/L $\text{Ni(NO}_3)_2$ and 0.075 mol/L NaNO_3 dissolved in a mixed solvent (ethanol-water 50 %:50 % vol.) to determine the effect of heat treatment time and temperature on the capacitive properties of the formed electrodes.

An analysis of the sources [21–27] showed that there were no systematic measurements of the deposition process efficiency, especially at high concentrations. Moreover, no information was found regarding the effect on the electrochemical and electrochromic properties of films formed at increased concentrations.

3. The aim and objectives of the study

The study aimed to determine the efficiency of the processes of nickel hydroxide electrodeposition from concentrated nickel nitrate solutions.

The aim was to solve a number of problems:

- to determine the current efficiency and structural characteristics of nickel (II) hydroxides obtained at high concentrations of nickel nitrate (concentration range of 0.25–2 mol/L);
- to determine the electrochemical and electrochromic characteristics of the resulting nickel (II) hydroxide deposit from solutions with increased concentration.

4. Research materials and methods

4.1. Obtaining deposits to determine the structural characteristics and current efficiency (CE)

Nickel hydroxide was obtained from pure solutions of nickel nitrate in a cell separated by a partition. The cathode area was filled with a nickel nitrate solution, the anode area – with a potassium nitrate solution. Based on the calculation of a sufficient amount of nickel hydroxide to ensure X-ray phase analysis and calculation of the current efficiency by the cathode weight gain, the deposition was carried out on a cathode with large dimensions (100×100 mm). The deposition conditions are given in Table 1.

After deposition, the cathode was rinsed in distilled water, dried in air at room temperature, and weighed on an analytical balance. The theoretical calculation of the deposited hydroxide was carried out according to Faraday's law by reactions (1) and (2). The structure was determined on a DRON-3 X-ray diffractometer in Co- K_{α} radiation.

Parameters of obtaining Ni(OH)₂ for X-ray phase analysis

No.	Cathodic space $C_m(\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$, mol/L	Anodic space $C_m(\text{KNO}_3)$, mol/L	Current density, mA/cm ²	Deposition duration, h
1	0.25	0.25	0.625	7
2	0.5	0.5		
3	0.75	0.75		
4	1	1		
5	1.5	1.5		
6	2	2		

4.2. Formation of deposits to obtain electrochromic and electrochemical characteristics

The preparation of the substrate for coating included the removal of contaminants from the electrically conductive surface in several steps. First, the substrates were degreased and wiped with a paste of Na₂SO₄ and water. This was followed by sequential rinsing with running and distilled water, as well as treatment in 96 % ethanol.

The formation of Ni(OH)₂ polyvinyl alcohol (PVA) deposits was carried out by the cathodic template method in the following mode:

- substrate: polished nickel, 2×2 cm;
- solution of 1.5 and 2 mol/L Ni(NO₃)₂, 5 % PVA;
- deposition on the cathode with a current density of 0.625 mA/cm²;
- deposition duration 80 min.

After electrodeposition, the samples were rinsed in distilled water to remove residual water-soluble salts at 30 °C for 10 minutes and dried at room temperature for one day.

4.2. Electrochromic and electrochemical characteristics

The electrochemical characteristics were estimated from cyclic volt-ampere curves using a three-electrode scheme in a potential sweep mode from +201 to +751 mV relative to a normal hydrogen electrode at a rate of 1 mV/s. A silver chloride electrode in saturated potassium chloride was used as a reference electrode. The cell was made of transparent polymethyl methacrylate. Nickel foil served as a counter electrode. Along with determining the electrochemical characteristics of the electrodes, changes in film transparency were also recorded.

The setup for determining the electrochromic and electrochemical characteristics consisted of a light source (5,500 K, China), a transparent cell with an electrolyte (0.1 mol/L KOH), recording photoresistor, an electronic potentiostat (Elins R-8, Russia), an ADC (E-154, Russia) and a stabilized power supply unit.

The setup worked as follows: when the potential set by the potentiostat was changed, the color of the electrode changed. The light fell on the electrode from the light source, which was reflected from the shiny substrate and hit the photoresistor. The change in the color of the film on the electrode changed the reflected amount of light incident on the photoresistor. As a result, the resistance of the photoresistor changed, as well as the voltage drop across it. In turn, the voltage drop across the photoresistor was recorded by the program supplied with the ADC [28].

The specific capacity of the anodic and cathodic processes was determined by numerical integration of cyclic current-voltage curves in time by the trapezoidal method.

Table 1

5. Results of electrochemical and electrochromic characteristics of the Ni(OH)₂-PVA deposit

5.1. Current efficiency and structural characteristics of the deposits

To accurately determine the efficiency of the process, as well as the possibility of conducting X-ray phase analysis, a sufficient amount of nickel hydroxide was needed. To obtain such an amount of nickel hydroxide deposit, the formation was carried out for a long time on a cathode with a large area (7 hours, 95 cm²). It should be noted that, since solutions with polyvinyl alcohol are quite viscous and can remain in significant quantities on the deposit and, thereby, introduce a significant error, it was decided to remove this component from the electrolyte composition. As a result, the dependence of the current efficiency (CE) was obtained at a fixed current density in a wide range of concentrations, which is shown in Fig. 1.

The resulting dependence shows a non-linear dependence of CE on the nitrate concentration. It should be noted that the deposit weight gain was not recorded at points 1.5 and 2 mol/L Ni(NO₃)₂.

Obviously, in a pure nickel nitrate solution, the CE parameter sharply decreases to zero. The decrease in CE – $C_m(\text{Ni}(\text{NO}_3)_2)$ was exponential.

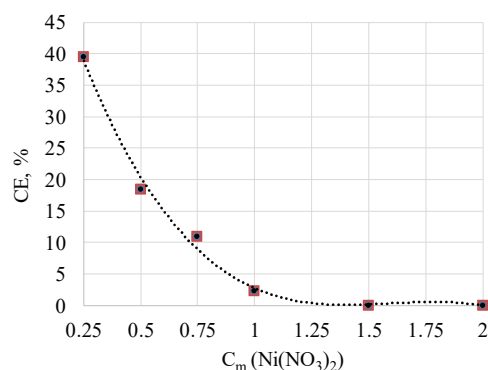


Fig. 1. Dependence of the current efficiency (CE) of nickel hydroxide at a cathode current density of 0.625 mA/cm² on the molar concentration of Ni(NO₃)₂

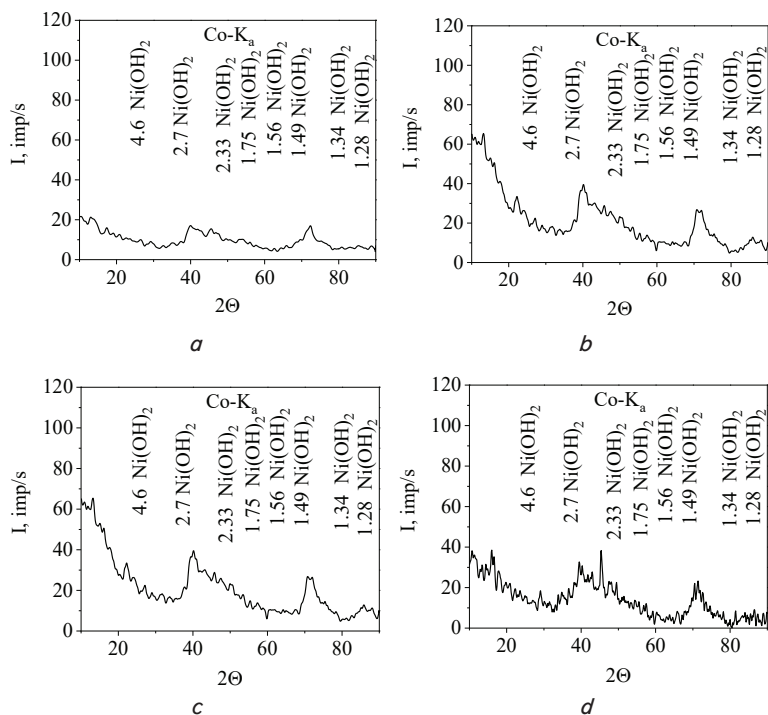


Fig. 2. Results of X-ray phase analysis for the nickel hydroxide deposit at a current density of 0.625 mA/cm² and molar concentration of Ni(NO₃)₂: a – 0.25 mol/L; b – 0.5 mol/L; c – 0.75 mol/L; d – 1 mol/L

X-ray diffraction patterns for nickel hydroxides obtained at different concentrations are shown in Fig. 2. For all hydroxides, two identical features can be distinguished. The first is the presence of distinguishable characteristic peaks. The second is the presence of an X-ray amorphous component. At the same time, no clear correlation was found between the characteristics of the diffraction patterns and the Ni(NO₃)₂ concentration used for the deposition.

Since the wavelength of the X-ray radiation used was Co-K_α=0.179 nM, based on the X-ray amorphousness of the diffraction patterns, the block size of an ideal crystal in a polycrystal was equal to or less than this value.

5.2. Electrochemical and electrochromic characteristics of deposits

The electrochromic and electrochemical characteristics for the deposit obtained from dilute solutions of Ni(NO₃)₂ (up to 1 mol/L) without and with the addition of water-soluble polymers were studied in detail in [19–26, 28–30]. Therefore, the deposit obtained at high concentrations above 1 mol/L Ni(NO₃)₂ was of interest. Although the CE value for Ni(OH)₂ tended to zero during deposition from pure concentrated solutions of Ni(NO₃)₂, an attempt was made to deposit from two solutions with a concentration of 1.5 and 2 mol/L. Coloring-bleaching curves, cyclic voltammetry curves (CVA) for these samples are shown in Fig. 3.

It should be noted that the surface of a nickel substrate always contains some oxides and hydroxides [34]. Therefore, to eliminate the influence of this factor, the nickel substrate was also cycled for determining the activity of surface oxide-hydroxide nickel compounds. The results showed an almost complete absence of the electrochemical activity of these oxides, which is most likely due to their small amount.

Analysis of the data presented in Fig. 3 suggests that the deposits obtained from concentrated solutions of Ni(NO₃)₂ with the addition of PVA exhibit electrochromic and electrochemical activity. In this case, the electrochromic characteristics are somewhat worse than those from concentrated solutions [18]. Although during prolonged deposition from a pure solution of 1.5 and 2 mol/L Ni(NO₃)₂, the cathode weight gain was not recorded (Fig. 1), during deposition from a solution with PVA, nickel hydroxide was formed (Fig. 3, b, d).

The optical characteristics of the films prepared from 1.5 mol/L Ni(NO₃)₂ and 2 mol/L Ni(NO₃)₂ are similar in shape and transparency (*T*, %) in the colored and bleached state. The average coloration depth (*D*, %) defined as the difference between the transparency (*T*, %) in a completely bleached and colored state for five cycles (Fig. 3, a, b), was practically the same (Fig. 4, a).

At the same time, the cathode capacity of the films was somewhat different (Fig. 4, b). Moreover, the capacity was lower for the sample obtained from a more concentrated solution.

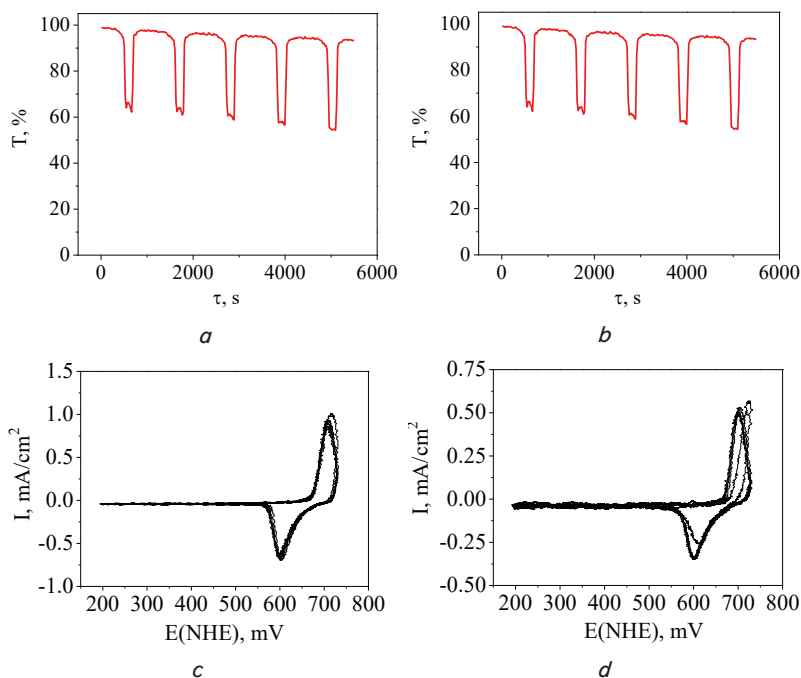


Fig. 3. Results for nickel hydroxide deposits at high concentrations of Ni(NO₃)₂: a – coloring-bleaching curve for the deposit obtained from 1.5 mol/L Ni(NO₃)₂; b – coloring-bleaching curve for the deposit obtained from 2 mol/L Ni(NO₃)₂; c – CVA for the deposit obtained from 1.5 mol/L Ni(NO₃)₂; d – CVA for the deposit obtained from 2 mol/L Ni(NO₃)₂

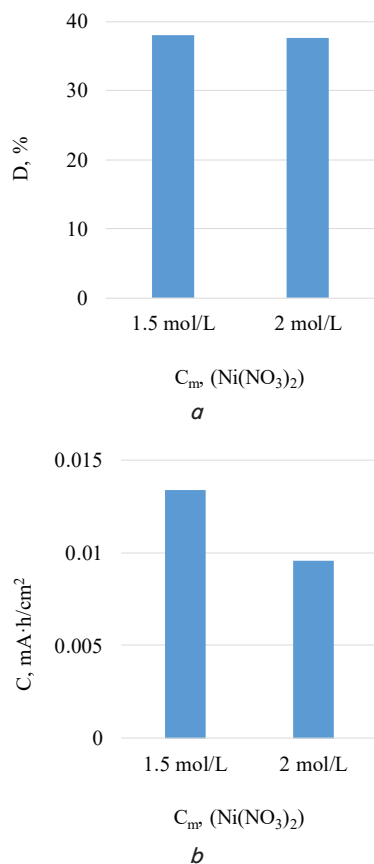


Fig. 4. Results for nickel hydroxide deposits obtained at high concentrations of Ni(NO₃)₂: *a* – average coloration depth for films obtained from 1.5 and 2 mol/L Ni(NO₃)₂; *b* – average cathode capacity of films obtained from 1.5 and 2 mol/L Ni(NO₃)₂ (determined from CVA, Fig. 3, *c*, *d*)

6. Discussion of the results of studying the characteristics of deposits obtained at high concentrations

Obtaining nickel hydroxide directly on a substrate is of practical importance since it can be used to achieve various purposes: formation of electrodes for chemical current sources, electrochromic elements, sensors, or electrodes of various electrochemical devices. Therefore, it is important to know the efficiency of the deposit formation process.

In the case of nickel hydroxides formed from a series of pure nitrate solutions in the range of 0.25–2 mol/L, the process efficiency decreased with an increase in the Ni(NO₃)₂ concentration. In this case, the CE of the process reached 0 at a concentration of 1.5 and 2 mol/L Ni(NO₃)₂ (Fig. 1). However, in the presence of PVA, the efficiency of the process increased, since the deposit obtained from solutions containing the polymer and 1.5, 2 mol/L Ni(NO₃)₂ exhibited electrochemical and electrochromic activity (Fig. 3).

This behavior may probably be due to a decrease in the diffusion coefficient in viscous solutions, which, upon prolonged deposition, will lead to a decrease in the near-electrode concentration of Ni²⁺ and NO₃²⁻ ions. Thus, the actual concentration will shift to the region of lower concentrations and the CE of the process will increase according to the dependence obtained in Fig. 1.

An unresolved and interesting issue is the increase in the efficiency of the electrodeposition process with the addition of PVA. In [32], the utilization rate of hydroxide in thin-film electrodes based on the Ni(OH)₂-PVA composite was shown to be about 100 %. This means that the active substance is electrochemically active throughout the entire thickness. Taking into account these data, it is possible to calculate the CE of the deposition process in the presence of PVA using the previously determined cathode capacities (Fig. 4, *b*).

It is worth noting that it is possible to calculate CE for concentrated solutions of Ni(NO₃)₂ with the addition of PVA only indirectly. This is because, on the one hand, the Ni(OH)₂ weight gain under these conditions can be distorted by the presence of PVA in the deposit. On the other hand, if we estimate the weight gain based on the already obtained indirect measurements of CE, then it will be 0.18 mg at CE=3.2 %, which is at the sensitivity limit of the analytical balance. Thus, the indirect estimation of CE from the capacitive characteristics can become a powerful tool for determining the trace amounts of deposited substances.

This calculation was based on the one-electron reduction reaction of NiOOH in Ni(OH)₂ (Fig. 5). Therefore, the current efficiency increased from zero to 3.2 % and 2.3 % at 1.5 and 2 mol/L Ni(NO₃)₂, respectively.

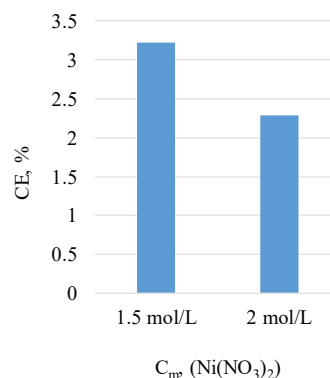


Fig. 5. Current efficiency for nickel hydroxide deposit obtained at high Ni(NO₃)₂ concentrations

Optimization of the processes for obtaining nickel hydroxide compounds consists in finding the optimal parameters for each case. In the case of electrochromic elements during optimization in [33], it was shown that the reversible operation of electrochromic films occurred at temperatures up to 40 °C. It was also determined that after holding for 8 hours in the working solution at temperatures of 40–60 °C, a reversible deterioration occurred, and above 70 °C, irreversible deterioration of electrochromic and electrochemical characteristics. This is important for these devices as they will be subject to cyclic heating and cooling. Similar experiments should be carried out for each specific application of nickel hydroxides, for example, if they are used as precursors to form electrodes on which hydrogen evolution is carried out [34]. In this case, further development of the preparation of multifunctional nickel hydroxide films based can be the production of mesoporous deposits using surfactants [35]. The pore nano-size will improve electrochemical and other functional characteristics by improving the supply (removal) of ions involved in the reactions.

Based on the results obtained in this work, it can be recommended as a starting condition not to exceed the concentration of 0.5 mol/L Ni(NO₃)₂ during the electrochemical

deposition of nickel hydroxide, and to use the PVA additive in the electrolyte at high $\text{Ni}(\text{NO}_3)_2$ concentrations (Fig. 1, 5).

7. Conclusions

1. The current efficiency of the electrodeposition process from a $\text{Ni}(\text{NO}_3)_2$ solution in the range of 0.25–

2 mol/L has decreased from 41 % to 0 even at a concentration of 1.5 mol/L, and the dependence has an exponential form.

2. The use of polyvinyl alcohol in the electrodeposition solution has led to an increase in the process CE from 0 % to 3.2 and 2.3 % at 1.5 and 2 mol/L $\text{Ni}(\text{NO}_3)_2$, respectively. It has been shown that PVA affects the mechanism of electrochemical deposition of $\text{Ni}(\text{OH})_2$.

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