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DETERMINATION OF THE EFFECT OF EXPOSURE CONDUCTED IN KOH SOLUTIONS AT DIFFERENT TEMPERATURES ON THE PROPERTIES OF ELECTROCHROMIC Ni(OH)₂-PVA FILMS

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To determine the effect of exposure of film composite electrodes based on Ni(OH)₂-polyvinyl alcohol to an alkaline solution at high temperatures on the electrochromic and electrochemical characteristics, a series of films was obtained. The films were obtained on a glass substrate coated with fluorine-doped tin oxide. The coating of the substrates was carried out by the cathodic template method under the same conditions. The resulting precipitates were treated by keeping them in an alkali solution at different temperatures: 30, 40, 50, 60, and 70 °C for 8 hours, thereby simulating the operating conditions of an electrochromic device in a hot climate.

It was found that the exposure temperature directly affected the electrochemical and electrochromic properties of the treated films. In this case, the cyclic volt-ampere curves showed a decrease in the peak values of the current densities and a lower rate of establishment of characteristics with an increase in the treatment temperature. At a maximum treatment temperature of 70 °C, the properties of the film significantly changed towards deterioration.

According to the results of the experiments, three temperature ranges of treatment were identified. The first one was in the range up to 40 °C, in which the films showed significant electrochromic and electrochemical activity after treatment. The second interval was between 40 and 60 °C, in which the coatings showed a reversible deterioration in electrochromic and electrochemical activity. After treatment in the second interval, the films gradually restored their performance during electrochemical cycling. The third interval was from 70 °C and above. The films treated in this temperature range irreversibly lost their electrochemical and electrochromic activity.

The study also proposed mechanisms to explain changes in the characteristics of electrodes during treatment, as well as possible ways to combat temperature degradation

Keywords: electrochromism, electrodeposition, nickel hydroxide, temperature, potassium hydroxide, recrystallization, aging, degradation

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1. Introduction

Energy-saving technologies are one of the options for reducing the negative impact of humanity on the planet. When using such technologies, the consumption of energy

of various types, including electrical energy, is reduced. In turn, the saving of electrical energy allows reducing the consumption of fossil energy carriers, thus, reducing the environmental pressure on nature, as well as greenhouse gas emissions [1].

New architectural trends that lead to the creation of premises with a so-called “open space” layout and a large percentage of the glazed surface of buildings lead to the fact that the development of variable-transparency windows is becoming relevant [2]. Such windows, called “smart”, are already used, but not widely due to the high price. Despite the mentioned drawback, simulations show that the use of “smart” windows can significantly reduce electricity consumption. Also, their use reduces electricity consumption during peak periods of consumption and also allows saving electricity needed for premises lighting [3].

Changes in the optical characteristics of “smart” windows are implemented using systems with suspended particles [4], liquid crystal systems [5], as well as electrochemical electrochromic materials [6]. The latter are industrially used in variable-transparency windows. Despite the wide range of electrochemical electrochromic materials being developed, inorganic compounds are currently used, mainly oxide or hydroxide compounds of metals [7]. One of these compounds is nickel (II) hydroxide. The study of the characteristics of thin films of nickel hydroxide, new methods of $\text{Ni}(\text{OH})_2$ deposition in the framework of improving the operating parameters of “smart” windows is an important and urgent research issue.

2. Literature review and problem statement

The biggest obstacle to the introduction of variable-transparency windows is their high price, which, among other things, is determined by the deposition of films of the finished device using energy-consuming methods. In [8], an electrochemical deposition method in the presence of polyvinyl alcohol was proposed. This method is cheap and also produces high-quality films with good adhesion to the substrate.

To improve the quality of such films, an electrochemical surface treatment with shallow etching of the electrically conductive part of the substrate was proposed in [9], and in [10] the positive effect of laser treatment of already formed films was shown.

When the coating is operating in a real electrochromic device, voltages may arise at the substrate-film interface due to cyclic heating and cooling. It should also be noted that research is being carried out in the direction of measuring characteristics at different temperatures. For example, in [11], which was carried out on a real prototype of an electrochromic device, some critical test parameters were identified. Among them, the most important ones were thermal cycling, UV resistance, heat storage, and thermal shock.

In [12], the optimization of the switching voltage between the colored and uncolored states of the electrochromic device at elevated temperatures was carried out. It was noted that the surface of the electrochromic device could reach 70 °C and the operating voltage changed significantly. Also, the authors found that the correct choice of the operating voltage of the electrochromic device was critical for the durability of the device. The researchers concluded that since high temperatures require less voltage, it is necessary to take into account the temperature change of this value, including in the algorithms for powering the electrochromic device.

In [13], it was shown that temperature differences in the range from +10 to +30 °C adversely affected the adhesion of the film to the substrate in some cases. The authors also proposed to use pretreatment of the substrate by etching to increase the adhesion of electrochromic films.

It was shown in [14] that exposure of electrochromic composite $\text{Ni}(\text{OH})_2$ -polyvinyl alcohol (PVA) films to a working solution (0.1 M KOH) and different media at a temperature of 80 °C affected further operational properties. Thus, the films exposed to air at 80 °C showed a slight increase in characteristics. On the contrary, exposure to alkali at the same temperature led to irreversible losses of electrochromic and electrochemical activity.

The researchers in [15] studied the characteristics of a material related to nickel hydroxide – NiO. Electrochromic films were obtained by the sol-gel method by immersing substrates. The next step was to anneal the film at an elevated temperature in the range from 250 to 450 °C. It was shown that the treatment of coatings at relatively low temperatures (300 °C) gave optimal results.

Electrochromic devices containing WO_3 , NiO films, and an electrolyte based on LiClO_4 dissolved in propylene carbonate [16] were tested at different temperatures. The researchers found that as the operating temperature rises from –40 °C to +80 °C, the electrochromic properties of the device generally improved. At the same time, signs of degradation were found during the operation of the electrochromic cell at above 60 °C.

Nickel hydroxide is the basis for the positive electrode of alkaline batteries [17]. The negative effect of high temperatures on the electrochemical characteristics is also manifested in this type of device, as shown in [18]. To reduce the degradation of the active battery material, the authors proposed to cover $\text{Ni}(\text{OH})_2$ with a layer of $\text{Co}(\text{OH})_2$ doped with calcium.

The reason for the deterioration of characteristics is the process of spontaneous recrystallization of $\text{Ni}(\text{OH})_2$. This process is known as aging [19].

Analysis of the above information demonstrates that the degradation of $\text{Ni}(\text{OH})_2$ films in an alkaline medium has been studied only partially, and questions remain that require additional research.

3. The aim and objectives of the study

The study aimed to determine the effect of exposure to a working solution at elevated temperatures on the electrochromic and electrochemical characteristics of $\text{Ni}(\text{OH})_2$ -PVA films. Achievement of this goal will allow predicting the behavior of the finished “smart” window in real operating conditions.

Within the framework of this aim, the following research objective was formulated:

- to determine changes and dynamics of changes in the electrochromic and electrochemical characteristics of the films after exposure.

4. Materials and research methods

Deposition of $\text{Ni}(\text{OH})_2$ -PVA coatings.

All $\text{Ni}(\text{OH})_2$ -PVA films were obtained on a 0.9 mm thick glass coated with a conductive transparent oxide –

SnO₂ with the addition of fluorine (hereinafter referred to as FTO glass). The specific surface resistance of the FTO glasses was less than 10 Ohm/sq. (Zhuhai Kaivo Optoelectronic Technology Co. Ltd. (China)).

The preparation of the substrate for the coating included degreasing and wiping with a paste of soda ash and water. This was followed by sequential rinsing with running and distilled water, as well as ultrasound radiation treatment in 96 % ethanol: duration – 10 min, power – 60 W, radiation frequency – 41,500 Hz. After that, the glass was dried, and immediately before electrodeposition was wiped with a lint-free cloth soaked in 96 % ethanol. The working area was 4 cm².

The Ni(OH)₂-PVA deposits were formed by the cathodic template method in the following mode:

– 0.01 M Ni(NO₃)₂, 4 % PVA solution;

– deposition on the cathode with a current density of 0.1 mA/cm² for 10 minutes.

After the coating was formed, the electrode was rinsed from the electrolyte in distilled water at 30 °C for 10 minutes. Drying of the obtained precipitates was carried out at room temperature for one day.

Exposure of precipitates at elevated temperatures.

The films obtained under the same conditions were kept in a 0.1 M KOH solution at 30, 40, 50, 60, 70 °C for 8 hours. Then the electrode was rinsed with distilled water and the characteristics were determined.

Electrochemical and optical tests.

To assess the electrochemical and optical characteristics, a setup was used, which included: a light source – LED (5500 K, China); transparent cell with electrolyte (0.1 M KOH), recording photoresistor, electronic potentiostat (Elins R-8, Russia), ADC (E-154, Russia) and power supply unit. The setup worked as follows – when the potential was changed, which was set using a potentiostat, the color of the electrode changed. At the same time, the light passed through it, which fell on the photoresistor. When the transparency of the electrode changed, the illumination of the photoresistor and, as a consequence, its resistance, as well as the voltage drop, changed. In turn, the voltage drop across the photoresistor was recorded by the program supplied with the ADC [14].

Assessment of electrochemical characteristics was carried out by the method of potentiodynamic cycling according to a three-electrode scheme in the mode [+201; +751 mV] relative to the NHE at $v=1$ mV/s. A silver chloride electrode in saturated potassium chloride was used as a reference electrode. The cell was made of transparent polymethyl methacrylate (PMMA). Nickel foil served as a counter electrode. During cycling, in parallel with the electrochemical characteristics of the electrodes, changes in the transparency of the film were also recorded.

The specific capacity of the anodic and cathodic processes was determined by the numerical integration of cyclic current-voltage curves over time by the trapezoidal method. Average specific capacities were calculated as the arithmetic mean of values over five cycles.

Visual assessment of electrodes.

To compare the quality of the films after all tests, the electrodes were colored by sweeping the potential of the film electrode from +201 to +751 mV relative to the NHE at $v=1$ mV/s. Then photographs were taken from different angles.

5. Results of studying the characteristics of thin-film electrodes treated at different temperatures

Fig. 1–5 show the results of the potentiodynamic cycling of the electrodes with the recording of transparency changes. The cyclic voltammetry curves (CVA) of the treated electrodes differ significantly in appearance.

If we analyze each of the CVA, the curves have three identical features. The first is the peak of the anodic current, which is characterized by the reactions of oxidation of hydroxide to oxyhydroxide (1):



The second is the rise in the most positive potential region, which indicates the release of oxygen according to reaction (2):



The third is the peak of the cathodic current, which describes the process of reduction of NiOOH to Ni(OH)₂, corresponding to the reverse reaction (1).

Analysis and comparison of CVA give the following picture. The heights of the peaks decrease with an increase in the alkali exposure temperature. In addition, at 70 °C, the film oxidation and reduction peaks virtually disappear. The peaks reach constant values of heights and positions in the samples treated at relatively low temperatures. On the other hand, the difference between the potential peaks E_a and E_k for the samples decreases with increasing temperature in the series 30 – 40 – 50 – 60 – 70 °C as follows 109 – 118 – 93 – 91 – 37 mV. Moreover, the difference decreases mainly due to the positive shift of the cathodic peak. In addition, we can say that the shape of the curves also changes. This is especially evident for the sample held at 70 °C. It is obvious that treatment at elevated temperatures significantly affects the electrochemical characteristics of the films.

The same trends can be seen in the transparency curves. In this case, the transparency value in percent (T, %) is plotted on the ordinate axis, where 100 % is the total transparency of the coated glass and 0 % is the completely opaque coating.

The transition to the stationary transparency value in the colored state (the lower points of the curves) is faster for the samples treated at 30 and 40 °C. For example, already from the second cycle, the transparency of the sample treated at 30 °C practically does not change (T): 43 % (1st cycle) → 9 % (2nd cycle) → 7 % (3^d cycle) → 7 % (4th cycle) → 8 % (5th cycle). Further, with increasing temperature, the decrease in transparency occurs more slowly. For the film treated at 60 °C, the transparency in the colored state changes as follows (T): 91 % (1st cycle) → 68 % (2nd cycle) → 47 % (3^d cycle) → 34 % (4th cycle) → 26 % (5th cycle).

Special attention should be paid to the shape of the characteristic of the transparency change of the sample treated at 70 °C, Fig. 5, b. The shape of the curve radically changes compared to the curves of other samples (30–40 °C, Fig. 1–4, b).

Analysis of the curves shows that the optical characteristics of the films generally correlate with the electrochemical characteristics. Moreover, there is a relationship between the exposure temperature and the electrode characteristics.

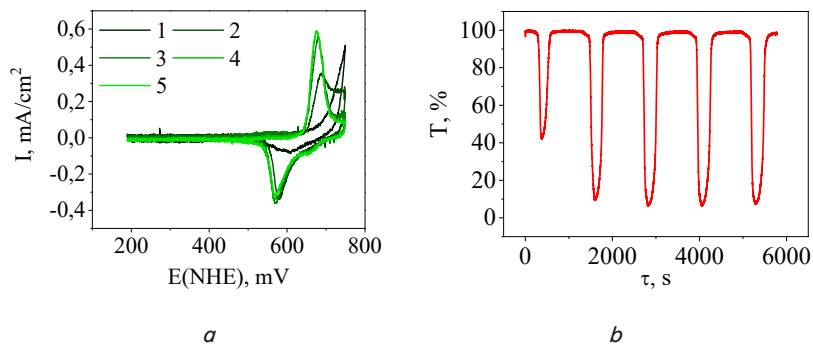


Fig. 1. Characteristics of the thin-film electrochromic electrode exposed to 0.1 M KOH at 30 °C for 8 hours: *a* – CVA; *b* – transparency curve after cycling

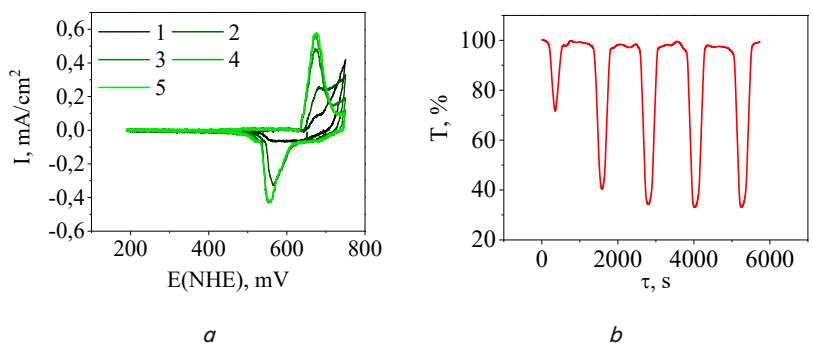


Fig. 2. Characteristics of the thin-film electrochromic electrode exposed to 0.1 M KOH at 40 °C for 8 hours: *a* – CVA; *b* – transparency curve after cycling

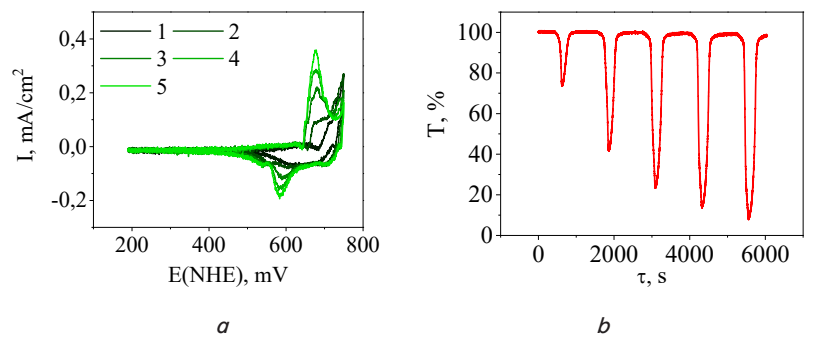


Fig. 3. Characteristics of the thin-film electrochromic electrode exposed to 0.1 M KOH at 50 °C for 8 hours: *a* – CVA; *b* – transparency curve after cycling

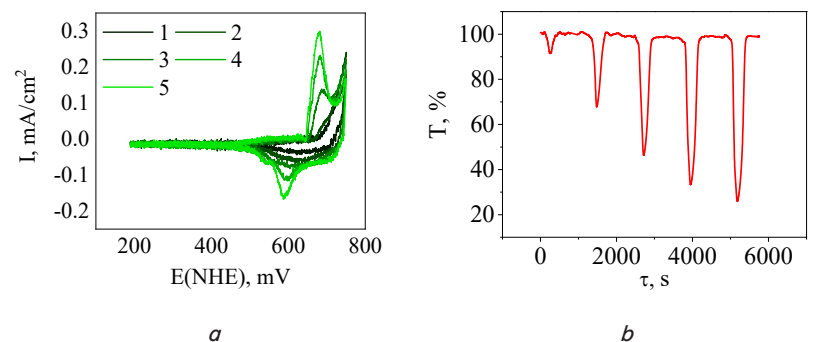


Fig. 4. Characteristics of the thin-film electrochromic electrode exposed to 0.1 M KOH at 60 °C for 8 hours: *a* – CVA; *b* – transparency curve after cycling

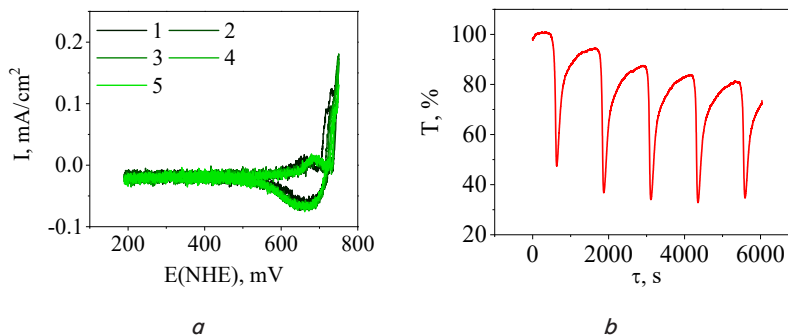


Fig. 5. Characteristics of the thin-film electrochromic electrode exposed to 0.1 M KOH at 70 °C for 8 hours: *a* – CVA; *b* – transparency curve after cycling

It should be noted that despite exposure to the solution, the films treated at 50 and 60 °C gradually return to normal operating parameters, which can be seen from the rise in the peak values and the increase in the coloration depth. The value of the coloration depth means the difference in transparency between the fully bleached and completely colored state for a given cycle, calculated from the transparency curve, i.e. $D = T[\text{bleached}] - T[\text{colored}]$, %. The worst electrochemical and optical characteristics are demonstrated by the film treated at 70 °C. Moreover, [14] shows that the treatment temperature of 80 °C of film electrodes based on Ni(OH)₂ leads to the irreversible loss of electrochromic characteristics. Considering that when cycling the electrode treated at 70 °C, the shape of the CVA and the coloration curve sharply changes, and there is no increase in peak currents during cycling, we can say the following. Most likely, a temperature above 70 °C irreversibly worsens the electrochromic qualities of the electrode, and above 80 °C, leads to their complete disappearance. This is also confirmed by photographs of the electrodes (Fig. 6). Analysis of the photos shows that the films have a good appearance for the treatment range of 30–60 °C. Treatment at 70 °C spoils the film, but the coating still has a small electrochromic activity. Taking into account that the characteristics of the films treated at 50 and 60 °C gradually increased, we can make a preliminary conclusion that a short-term (up to 8 hours) increase in temperature to 60 °C is acceptable. It should be said that this may be sufficient for work in hot countries, since maximum temperatures are usually observed from 12:00 to 16:00, i. e. about 4 hours. In subsequent experiments, it is necessary to find out the following:

- what will happen during repeated cycling of the electrode treated at 50 and 60 °C;
- whether repeated treatments will irreversibly degrade the electrode characteristics;
- whether it is possible to increase the continuous exposure to elevated temperatures (50 and 60 °C).

For additional analysis of the treatment effect, the characteristics of the electrodes were calculated: the average coloration depth (for 5 cycles) and the average specific capacities of the processes (cathodic and anodic) (Fig. 7).

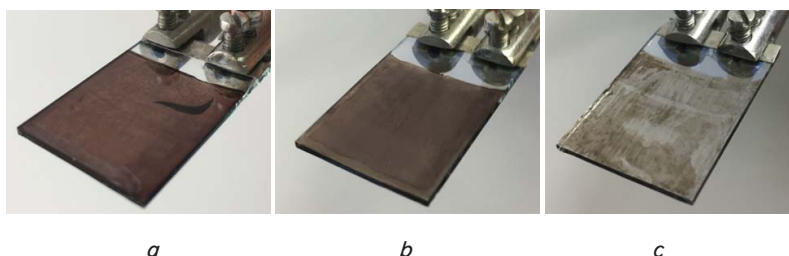


Fig. 6. Photographs of thin-film electrochromic electrodes: *a* – exposure to 0.1 M KOH at 30 °C for 8 hours; *b* – exposure to 0.1 M KOH at 60 °C for 8 hours; *c* – exposure to 0.1 M KOH at 70 °C for 8 hours

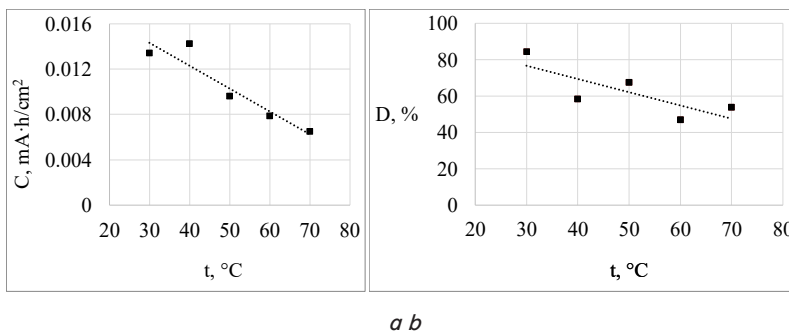


Fig. 7. Characteristics of electrodes from a series of experiments on exposure to 0.1 M KOH at different temperatures: *a* – average specific capacities of processes for 5 cycles (sum of cathodic and anodic ones); *b* – average coloration depths for 5 cycles

These characteristics also confirm the deterioration of the characteristics of the films when treated in alkali solutions at elevated temperatures.

6. Discussion of the results of studies of the characteristics of thin-film electrodes treated at different temperatures

The negative effect of elevated temperatures, above 30–40 °C, is obvious (Fig. 7). Such a change in characteristics is associated with the interaction of the film with the electrolyte solution [14]. Most likely, the process of deterioration of characteristics also takes place at low temperatures, but at high temperatures, it is sharply accelerated. Thus, [19] shows that nickel hydroxide powders, upon prolonged (up

to 2 months) exposure to concentrated alkali at room temperature, partially lost their electrochemical activity. This behavior of the films is caused by the process of recrystallization of the nickel hydroxide precipitate (aging process) through the dissolution of higher-energy crystals and the formation of lower-energy crystals. The formation is carried out with the material released due to the dissolved crystals. In this case, high-energy crystals are those that have crystal lattice defects, impurities, and nickel ions substituted in the crystal lattice with ions of other metals [20, 21]. The impurities can also include water molecules located in the defective zones and between the crystals.

It is known that α -Ni(OH)₂ [22, 23] contains water molecules (3α -Ni(OH)₂·2H₂O) in its structure, and this form is obtained by electrochemical deposition from solutions [24]. In this case, the α modification of nickel hydroxide is metastable and eventually passes first into β_{bc} -Ni(OH)₂ (badly crystalline) and into β -Ni(OH)₂ [25]. The latter is an additional factor of instability of the film characteristics at elevated temperatures used in the experiments. In addition, in Ni(OH)₂ powders, the surface-to-volume ratio of the particles is less than that of thin films (\approx 300 nm, [9]). Thus, in thin films, almost all the substance comes into contact with the solution, and the entire volume of nickel hydroxide will take part in the recrystallization process. Considering that zones and crystals with increased energy are also more electrochemically active, recrystallization will generally lead to a deterioration in the characteristics.

Thus, the fight against the deterioration of characteristics can be directed, on the one hand, to the maximum lowering of the operating temperature of electrochromic devices. On the other hand, based on the mechanisms described above, a logical step may be to increase the electro-

lyte viscosity or cover Ni(OH)₂ with other films. The latter should prevent the dissolution of the active material, but not interfere with the transfer of ions involved in the reaction.

At the same time, the limitation of this study is the short exposure at different temperatures compared to real films operating in “smart” windows. However, this limitation can be easily eliminated by additional research.

One of the ways to improve the characteristics of Ni(OH)₂ films in the context of aging can be deposition in the presence or addition of surfactants to the working electrolyte. These can be either existing surfactants used in the synthesis of Ni(OH)₂ (sodium dodecyl sulfate [26], dodecylbenzenesulfonate [27] cetrimonium bromide [28], synthanol ALM-10 [29]), or newly synthesized ones. For example, in [30], the product of the reaction of an oxyethylated surfactant (syntanol ALM-10) with adipic acid was synthesized, which can be used as a modifier of the morphology and properties of films based on Ni(OH)₂.

7. Conclusions

It was shown that the reversible operation of electrochromic Ni(OH)₂-PVA films is possible up to 40 °C. It was also determined that exposure to the working solution at 40–60 °C leads to a reversible, and above 70 °C, irreversible deterioration of electrochromic and electrochemical characteristics. In addition, the dynamics of changes in the electrochemical and optical characteristics depending on the exposure temperature is relatively smooth. Nevertheless, a visual assessment of the quality of the electrodes indicates a sharp deterioration in the appearance of the films upon exposure to 0.1 M KOH at a temperature of 70 °C.

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