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INVESTIGATION OF THE ELECTROCHROMIC PROPERTIES OF NI(OH)$_2$ FILMS ON GLASS WITH ITO-NI BILAYER COATING

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Електрохромні покриття були отримані на склі, покритому оксидом індій-олово (ITO) і склі з ITO і нанесеним на нього тонким шаром металевого нікелю. Нікель на поверхню покриття ITO наносили з розчину електрохімічного нікелювання. Режим нанесення металевого нікелю на шар ITO був наступний – катодна густина струму 0,5 А/дм^2, тривалість нанесення 3 с. Такий режим був обраний у зв'язку з тим, що при більшій тривалості нанесення та (або) густині струму, отримуване покриття із металевого нікелю ставало непрозорим. Дві зазначені основи використовували для нанесення катодним темплатним методом електрохромних плівок на основі Ni(OH)$_2$. В результаті було показано, що електрохромне покриття, отримане на склі з покриттям ITO-Ni, має кращі питомі характеристики ніж скло з ITO: більшу глибину затемнення, а також більші струми окислення-відновлення на циклічній вольтамперограмі. При цьому було показано, що плівка, яка має найкращі характеристики, навпаки має меншу оберненість процесу затемнення-освітлення. У зв'язку з отриманими результатами був запропонований механізм, який пояснює роль проміжного шару металу. Він полягає у тому, що нанесений шар металевого нікелю утворює додаткові електричні контакти між поверхнею субстрату та електрохромною плівкою. При цьому шар нікелю може допомагати утримувати шар електрохромної плівки, а під час нанесення позитивно впливає на розподілення струму. З іншої сторони, менша оберненість процесу затемнення-освітлення плівки на склі з покриттям ITO-Ni може бути пояснена поступовим окисленням нікелю у лужному середовищі. Разом з цим було показано, що нанесення металевого нікелю призводить до деякого зменшення прозорості підкладки

Ключові слова: електрохромізм, електроосадження, тонкі плівки, Ni(OH)$_2$, нікель, індій-олов'яний оксид, гідроксид нікелю

1. Introduction

In the 21st century, the city development is viewed through the design and creation of so-called "smart cities", in which the entire infrastructure is constituted by "smart devices" and "smart houses" connected to computer networks and Internet. The main purpose of "smart devices" is to optimize time for completion of various tasks, reduce resource consumption and increase comfort [1, 2]. One of such devices are smart windows. These devices employ various systems in order to alter their optical properties: color, opacity, reflectivity. The application range of "smart windows" is rather broad. Creation of suitable lighting, inclusion of elements that dynamically change color into the interior design, protection of museum showpieces from bright light, patient examination rooms in hospitals, improvement of drivers' safety – is far from complete list of application for "smart windows". Additionally, the "smart windows" can be used as information displays that show currency exchange rate, prices, weather. The main advantage of using such devices indoors is lower power consumption for conditioning up to 49 %, decreased load on the power grid in peak hours up to 16 % and cost savings on light up to 51 % [3].

Electrochemical systems are viewed as possible candidates for active elements of the smart window responsible for the change of optical properties. The main advantage of these systems is that they don't require additional electric current to sustain colored (bleach) state. Usually, the electric current is only consumed when the electrode reaction occurs, which results in the colored material.

Nickel hydroxide is one of such materials that possess electrochromic properties and can find application in "smart windows". The advantages of this material are, on the one hand, good specific electrochromic characteristics and, on the other ease and multitude of film deposition techniques and also relatively low cost of the resulting film. It should be noted that the cost of 1 m^2 of electrochromic film based on liquid crystals, on average, is about 200 USD, while the nickel-based electrochromic film would cost approximately 10 times cheaper.

The main problem of this material is low adhesion of $Ni(OH)_2$ film to the transparent conductive substrate and gradual degradation of the film itself [4, 5].

The solution to this problem can be a search of new deposition methods or application of combined or modified materials based on $Ni(OH)_2$ [6], and also modification of the material on which electrochrome is to be deposited.

2. Literature review and problem statement

Nickel hydroxide is a compound with a broad spectrum of applications. It is used for the creation of high-capacity accumulators [7, 8], in electrodes of asymmetric supercapacitors [9, 10], for the oxidation of some organic compounds [11, 12], and as a component of electrodes for water decomposition [13, 14].

Nickel hydroxide prepared in special conditions can be used as an electrochromic layer, which can reversibly change its optical characteristics between transparent and dark-brown color [15, 16] (1):

$$Ni(OH)_2 \leftrightarrow NiOOH + H^+. \quad (1)$$

Thin $Ni(OH)_2$ films are transparent and NiOOH has dark-brown color.

Because one of the main problems of this material is low adhesion to the transparent conductive substrate, the continuous use of this material as an electrochrome is not possible. This leads to degradation of electrochromic properties of nickel hydroxide because of disruption of electrical contact between $Ni(OH)_2$ and the transparent conductive substrate. The modern research papers on electrochromism of $Ni(OH)_2$ are primarily focused on the creation of composite and modified materials [17, 18]. For instance, the authors of the paper [18] have demonstrated a method for preparing a composite electrochromic material, composed of reduced graphene oxide, metallic nickel particles and NiO. This composite possesses higher specific characteristics than the base material composed of NiO. The paper [19] describes the preparation of the electrochromic material with an inner layer of silver that acts as a conductor. This enables to exclude a separate conductive layer from the structure of the electrochromic element. Other authors [20] proposed a composite material composed of nanoparticles deposited onto ITO-coated glass. The composite particles contained the electrochromic material inside and were coated with indium oxide particles. In_2O_3 provided additional electric conductivity to the composite material, which had a positive effect on specific characteristics. An alternative approach to improve adhesion is the modification of the substrate onto which electrochrome is to be deposited. For instance, the paper [6] describes the electrochemical treatment of the transparent conductive substrate, in order to alter the morphology and wetting, which resulted in improvement of electrochemical properties of deposited $Ni(OH)_2$.

The substrate onto which the electrochromic layer is deposited is usually a layer of transparent conductive metal oxides. The most commonly used types of the transparent conductive oxide layer are: ITO (indium-tin oxide) and FTO (fluorine-doped tin oxide) [21, 22]. The resistivity of these conductive substrates is significantly higher than that of metals.

The high resistivity of ITO (FTO and other transparent conductors) can negatively impact the distribution of the deposition process during electrochemical deposition of the film. The same reason can also impact electrochromic characteristics of nickel hydroxide films because the current density can vary significantly in different points of the electrode. On the other hand, the morphology of ITO [23, 24] and FTO [25, 26] films is rather flat on macroscale and microscale bumps are within one to tens of nanometers. The latter can be the reason for low adhesion of hydroxide films to the surface of transparent materials coated with ITO and FTO, and is related to the low real contact area of deposited $Ni(OH)_2$ and the conductive substrate. Thus, in order to increase specific conductivity and develop the surface of ITO (FTO) coatings, it has been proposed to deposit a thin layer of nickel with satisfactory transparency. Considering that research of the published paper yielded no information regarding such substrate modification with the aim to improve the characteristics of electrochromic $Ni(OH)_2$ films, it has been decided to conduct necessary experiments.

3. The aim and objectives of the study

The aim of the work was to study the properties of electrochromic $Ni(OH)_2$ films on glass with bilayer ITO-Ni coating.

In the scope of the set aim, the following objectives were formulated:

– to deposit electrochromic films based on $Ni(OH)_2$ onto glass with bilayer ITO-Ni coating;

– to evaluate the influence of morphology of ITO and bilayer ITO-Ni on the morphology of electrochromic films;

– to conduct a comparison of electrochromic and electrochemical properties of prepared samples.

4. Materials and methods

Materials used in the research.

The initial substrates were glass slides 20×20×1.1 mm coated with ITO (China), Fig. 1. Sheet resistance was 20 Ω/sq.

Fig. 1. ITO-coated glass substrates

The substrates were coated with nickel electrochemically using dull nickel-plating electrolyte. Deposition conditions and electrolyte composition are listed in Table 1. The deposition was carried out in a cell with a large electrode spacing with a thick nickel foil used as the anode.

Deposition conditions and electrolyte composition for the deposition of the electrochromic film are listed in Table 2 [4–6, 15].

Table 1

Composition of dull nickel-plating electrolyte and deposition conditions

Electrolyte composition	pH	Electrolyte temperature, °C	Cathodic current density, A/dm^2	Deposition time, c
$NiSO_4 \cdot 7H_2O$ – 160 g/L $NiCl_2 \cdot 2H_2O$ – 40 g/L H_3BO_3 – 35 g/L $Na_2SO_4 \cdot 10H_2O$ – 70 g/L	5.5	20	0.5	3

Table 2

Electrolyte composition and deposition conditions for electrochromic film

Electrolyte composition (cathodic chamber)*	Electrolyte temperature, °C	Cathodic current density, mA/cm^2	Deposition time, min
$NiNO_3$ – 1 M PVA – 50 g/L	30	0.625	8

*Note: * – KNO_3 1 M solution was loaded into the anodic chamber*

The deposition was conducted in a cell with 2 chambers – anodic and cathodic, separated with a low-permeability diaphragm. Nickel foil was used as the anode.

As a result, two electrochromic films were prepared: on glass coated with ITO and on glass with bilayer ITO-Ni coating. For convenience, the samples were labeled as listed in Table 3.

Table 3

Sample labeling

Sample description	$Ni(OH)_2$ deposited on glass coated with ITO	$Ni(OH)_2$ deposited on glass coated with ITO with electrochemically deposited Ni
Label	ITO-NH	ITO-Ni-NH

Morphology analysis.

The morphology of PVA-$Ni(OH)_2$ composite films was analyzed using scanning electron microscope (SEM) JEOL JSM-6510LV (Japan).

Evaluation of electrochromic and electrochemical properties of prepared samples.

The optical and electrochemical properties of the samples were evaluated by means of cyclic voltamperometry (CVA) with simultaneous recording of the coloration-bleaching process. The tests were conducted in a cell presented in Fig. 2. Ag/AgCl (KCl sat.) was used as a reference electrode. Nickel foil was used as a counter-electrode. For all measurements, 0.1 M KOH solution was used as an electrolyte. Optical characteristics were recorded using the analog-to-digital converter ADC E-154 (Russia), electrochemical characteristics measurement was conducted using the digital potentiostat-galvanostat Ellins P-8 (Russia).

Cycling regime: potential window from +200 to +750 mV, scan rate 1mV/s, number of cycles – 5.

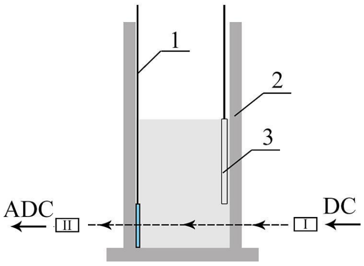


Fig. 2. Cell with the electrode for electrochemical and optical tests: 1 — Working electrode with the electrochromic film deposited onto ITO-coated glass; 2 — Frame made of transparent plastic; 3 — counter-electrode; I — source of white light (5,500 K); II — photoresistor. DC — source of stabilized voltage; ADC — analog-to-digital converter

5. 1. Morphology analysis of initial substrate and with deposited layer of metallic nickel

Based on preliminary tests, the following conditions for deposition of metallic nickel were chosen – cathodic current density 0.5 A/dm^2, deposition time 3 s. The values of deposition parameters are minimal, because high current density and long deposition time result in complete coverage of ITO with a mirror-like opaque layer of metal – Fig. 3.

Fig. 3. Photograph of the substrate (ITO-coated glass) in the holder, after deposition at a cathodic current density of 2 A/dm^2 for 20 seconds, till mirror-like shine (outlined in red)

Nickel was chosen as the substrate material because it would not poison the film even if oxidized during anodic polarization during coloration. Assuming uniform deposition, it is estimated that under given conditions, the thickness of nickel layer is about 5 nm (50 Å).

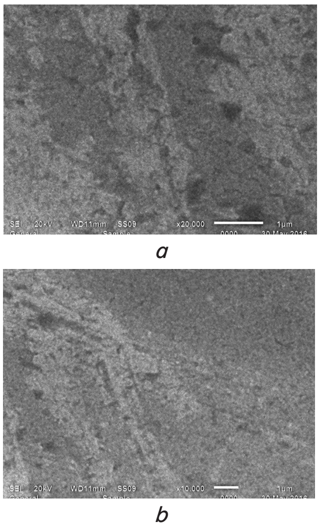
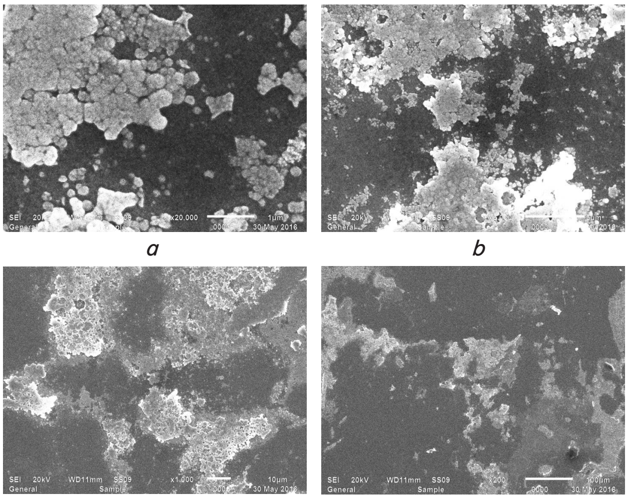


Fig. 4. SEM images of the ITO coating on glass: *a* — probe point № 1; *b* — probe point № 2 (magnification ×20,000)

Morphology of the initial substrate and the substrate with the deposited nickel layer is shown in Fig. 4 and 5 respectively.

It should be noted that the relief of the ITO surface is unpronounced and rather flat (Fig. 4), and the height of bumps varies from dozens to hundreds of nanometers.

At the same time, the surface of ITO coated with nickel (Fig. 5) shows a greater number of bumps. Clusters of nickel spheres can be observed, which don't cover the surface of ITO completely. Obviously, these clusters can act as conductive bridges between the substrate and the electrochromic layer during deposition of $Ni(OH)_2$.

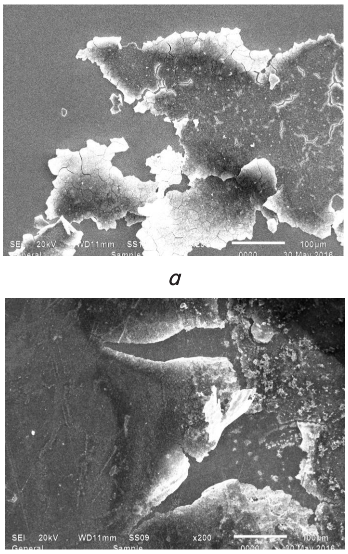


a *b*

c *d*

Fig. 5. SEM images of ITO with deposited nickel: *a* – magnification ×20000, *b* – magnification ×5,000, *c* – magnification ×1,000, *d* – magnification ×200

After the substrate with bilayer ITO-Ni coating was prepared, the electrochromic films were prepared onto both samples. Thus, 2 films on glass with ITO and on glass with bilayer ITO-Ni coating were prepared. Sample morphology images are shown in Fig. 6.



a

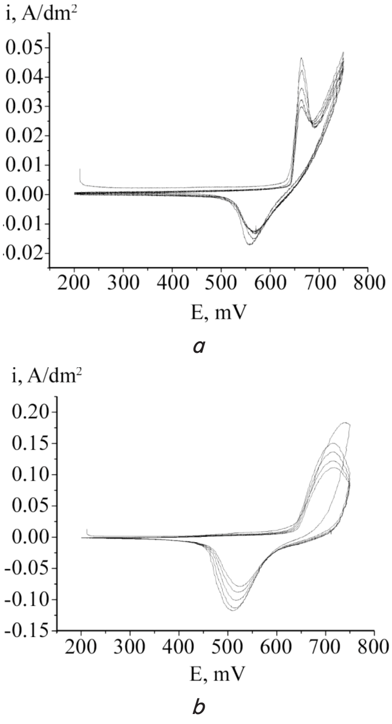
b

Fig. 6. SEM images of samples with deposited electrochromic films (magnification ×200): *a* – ITO-NH; *b* – ITO-Ni-NH

Morphology analysis allows drawing a few important conclusions. Electrochromic films deposited onto the substrate without nickel show many regions where the film either had not formed or peeled off and remained in the deposition cell. The electrochromic film that remained on the surface has a weak contact with the substrate. On the contrary, the film deposited onto glass with bilayer ITO-Ni coating shows better coverage, uniformity and contact with the substrate.

5. 2. Results of electrochemical and optical test of electrochromic films

As a result of the experiments, two electrochromic films were prepared. The deposition conditions were the same for both films, with the exception of substrate differences. Both films were subjected to potentiodynamic cycling with simultaneous recording of optical characteristics. Cyclic voltamperograms of both films are shown in Fig. 7, optical characteristics are shown in Fig. 8.



a

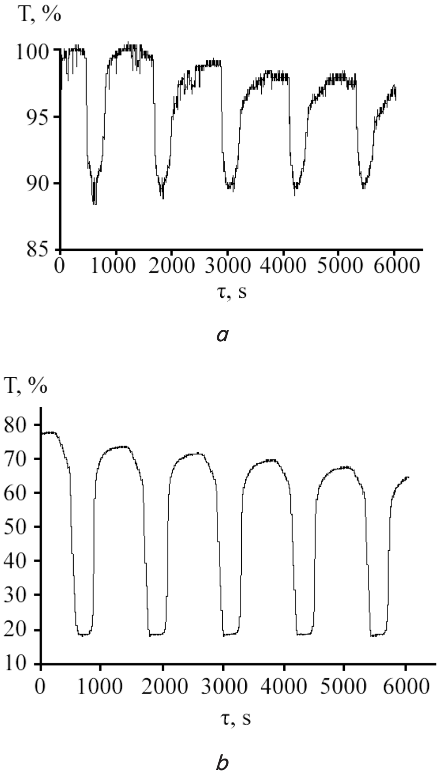
b

Fig. 7. Cyclic voltamperograms of electrochromic films: *a* – ITO-NH; *b* – ITO-Ni-NH

Comparison of cyclic voltamperograms revealed significant differences in electrochemical behavior. Thus, current densities for the ITO-Ni-NH film are about 4 times higher than those for the ITO-NH sample. In addition, the shapes of cyclic curves are different: for the ITO-NH sample the reduction and oxidation peaks are sharper and located at potentials of +570 and 670 mV respectively. For the ITO-Ni-NH sample, reduction and oxidation peaks are not as sharp and are located at potentials of +520 and +720 mV respectively. The difference between the peaks for ITO-NH is 100 mV and for ITO-Ni-NH is 200 mV. It should be noted that for the sample deposited on bare ITO, the oxygen evolution is clearly observed at potentials above the oxidation peak (Fig. 7).

Fig. 8 shows dependencies of film transparency (T) on time (τ) during potentiodynamic cycling. Comparison of optical characteristics of both films allows concluding that the film deposited onto the hybrid ITO-metal substrate (ITO-Ni-NH sample) possesses better characteristics. Thus, the coloration degree (difference between the bleached and colored state) is higher for the ITO-Ni-NH sample. However, as expected, the presence of even a thin layer of metal impacts transparency. The impact on transparency could even be observed upon initial observation the substrate with a thin layer of nickel appeared somewhat darker than the initial glass with ITO. Because of that, the initial point of the curve for ITO-Ni-NH lies below 100 %, at about 78 % – Fig 8, *b*. Additionally, the ITO-Ni-NH film shows an optical characteristic that is closer to the rectangular shape, but also shows significant irreversibility during bleaching.

The film deposited on the ITO substrate (ITO-NH sample) shows a lower coloration depth, but also shows better reversibility to the initial (bleached) state.



a

b

Fig. 8. Dependencies of transparency (T) on time for electrochromic films: *a* — ITO-NH; *b* — ITO-Ni-NH

The average coloration degree for the ITO-NH sample is 8 %, while for the ITO-Ni-NH sample the averaged coloration degree is 50 %, which is more than 6 times greater than that of the sample deposited onto bare ITO. Regarding reversibility (bleaching), the ITO-NH sample demonstrated better characteristics, with loss of initial transparency after cycling of 3 %. For the ITO-Ni-NH sample, the same value is about 10 %, which is more than 3 times greater than for the ITO-NH sample.

6. Discussion of morphology, electrochemical and optical characteristics of electrochromic films

The surface of the ITO substrate, obviously doesn't contain significant surface bumps and forms a poor contact with nickel hydroxide. Thus, the approach of creating microbumps by depositing intermediate conductive and transparent films can be a solution to this problem. In addition, low conductivity of the substrate, onto which the film is to be electrochemically deposited, can lead to non-uniform film formation. In turn, this can also be a reason for poor adhesion of the resulting films.

It is interesting that in reality a “web” of nickel clusters had formed instead of a uniform film of metallic nickel. Obviously, this is related to the known mechanism of electrochemical deposition of metals. In summary, this mechanism is described as follows: at the start of the deposition process, isolated metal “seeds” form, which then sprout covering the whole surface without the deposit. The further process occurs through the frontal growth of the deposit [27]. The formed nickel cluster could actively affect the electrochrome deposition process and also the coloration-bleaching process via the formation of conductive bridges from the conductive substrate.

The obtained results support previously proposed assumptions. It has been revealed that the films deposited onto the ITO-metal substrate have demonstrated better optical characteristics. It was also revealed that the peak current values on the cyclic voltamperogram of the ITO-Ni-NH sample are higher, which means the capacities under the curves on the voltammogram are also higher. In turn, the capacities characterized the amount of electricity used in oxidation and reduction of the film deposited onto the hybrid ITO-metal substrate. Nevertheless, the distance between the peaks for the ITO-Ni-NH sample is larger than for the ITO-NH sample, which indicated higher irreversibility. This is likely related to high transitional resistances of ITO↔Ni and Ni↔$Ni(OH)_2$.

An interesting fact is that, on the one hand, the characteristics of the ITO-Ni-NH sample are better than those of ITO-NH, but, on the other, the ITO-Ni-NH film shows higher irreversibility during bleaching. This fact probably indicates gradual destruction of conductive bridges, caused by oxidation of metallic nickel during anodic polarization in the basic medium.

Thus, it was found that modification of the substrate surface by deposition of a thin layer of nickel leads to a significant improvement of electrochromic and electrochemical properties of $Ni(OH)_2$ films. Also, an assumption can be made that for further improvement of surface modification, it is necessary to use metals that are more stable to anodic polarization instead of nickel.

7. Conclusions

1. As a result of analyzing deposition conditions for the deposition of a transparent metal layer onto ITO-coated glass from dull nickel plating electrolyte, the following optimal conditions were found – 0.5 A/dm^2, 3 s.

2. The morphology of the substrates and $Ni(OH)_2$ films deposited onto them have been studied. The analysis of SEM results has revealed that the deposited layer of metallic nickel was non-uniform and formed a “net” of spherical formations. The glass coated with ITO had a rather uniform surface with the bump height varying from dozens to hundreds of nanometers.

3. The comparison of characteristics of films deposited onto different substrates has revealed that surface modi-

fication of the ITO substrate by depositing a thin layer of nickel has a positive influence on electrochromic properties of films. The average coloration degree for the film deposited onto bilayer ITO-Ni coating was 50 %, which is more than 6 times higher than that of the sample deposited onto bare ITO. It was also revealed that even a thin layer of metallic nickel impacts the substrate transparency, which is a negative aspect of such approach.

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DETERMINIG THE PARAMETERS OF THE ACOUSTIC SYSTEM FOR THE PRIMARY TREATMENT OF WOOL

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Досліджено процес первинної обробки вовни з метою видалення з неї ґрунтових і гнойових забруднень, рослинних домішок і жиропоту. Первинна обробка вовни виконує ключову роль в збереженні природних властивостей вовняного волокна і дозволяє отримувати якісну сировину для текстильної промисловості. У процесі досліджень було встановлено, що для інтенсифікації процесів первинної обробки вовни необхідно використовувати акустичні коливання. Акустичні коливання діють на систему вовна-мийний розчин. Система вовна-мийний розчин знаходиться між решіточними транспортерами. Транспортери утворюють решітку з металевих стрижнів. Для визначення параметрів акустичної системи для первинної обробки вовни був проведений аналіз трьох задач. Задачі були пов'язані: з розсіюванням акустичних коливань на металевій решітці, на шарі вовна-мийний розчин і об'єднання рішень двох задач в одне за допомогою методу матриць розсіювання. Проведені дослідження дозволили визначити параметри системи для первинної обробки вовни акустичними коливаннями, що дозволяє досягти більшої ефективності в очищенні вовни в порівнянні з існуючими аналогами. В результаті теоретичних і експериментальних досліджень було встановлено, що промивка вовни повинна здійснюватися при наступних параметрах акустичних коливань в миючому розчині: частота звукового поля 1,1±0,1 кГц; інтенсивність звуку 1,1±0,01 Вт/см^2. При цьому товщина шару вовни на транспортері 0,06±0,01 м; швидкість руху транспортера 0,1 м/с; кількість перетворювачів на ванну 8±1 штук.

Застосування оптимальних параметрів в процесі безперервної промивки шерстяного волокна в водному розчині дозволяє отримати залишок жиру на шерсті в межах 1,5 % від кількості жиру немитої вовни при ГОСТ України до 2 %

Ключові слова: первинна обробка вовни, параметри акустичних коливань, коефіцієнт відбиття акустичної хвилі

1. Introduction

The main type of wool used in the woolen industry is sheep wool. It is used to produce a wide range of wool products for consumer and industrial purposes. The quality and cost of products made from wool mainly depend on the primary treatment of wool [1]. An analysis of the technological process and equipment used at factories during primary treatment of wool revealed their significant drawbacks: negative impact on the environment, energy-intensive production, materials consumption, large dimensions and mass, low quality of fibers [2]. Given this, there is a need to develop the