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RESEARCH ARTICLE

Reduction of hydrogen absorption into materials of membrane electrode assemblies in hydrogen generators

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Abstract

Objectives. To investigate the possibility of preventing hydrogen absorption into the functional structural materials of hydrogen-generating membrane electrode assemblies based on porous nickel, carbon black, and reduced graphene oxide with platinum–nickel and palladium–nickel nanoparticles.

Methods. The hydrogen absorption into materials of membrane electrode assemblies of alkaline electrolyzers was evaluated using an electrolyzer with variable temperature, reagent feed rate, and gas content.

Results. The study established the need to use reduced graphene oxide, in order to reduce hydrogen absorption and degradation of hydrogen-generating membrane electrode assemblies.

Conclusions. The service life test results and performance of the designed variants of prototypes of membrane electrode assemblies with nanostructured electrodes based on reduced graphene oxide, preventing hydrogen absorption into functional materials and their degradation, demonstrated the creation of hydrogen generators with high energy efficiency shows potential.

Keywords: *hydrogen generation, hydrogen absorption into metals, membrane electrode assemblies, nanocomposite electrodes, reduced graphene oxide, energy efficiency*

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НАУЧНАЯ СТАТЬЯ

Снижение наводораживания материалов мембранно-электродных блоков генераторов водорода

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Аннотация

Цели. Исследование возможности предотвращения наводораживания функциональных конструктивных материалов мембранно-электродных блоков генерации водорода на основе пористого никеля, сажи и восстановленного оксида графена, модифицированных наночастицами платина-никель и палладий-никель.

Методы. Для оценки степени наводораживания материалов мембранно-электродных блоков щелочных электролизеров была использована установка электролизера с возможностью контроля температуры, скорости подачи реагентов и содержания газов.

Результаты. Обоснована необходимость применения восстановленного оксида графена с целью снижения наводораживания и деградации мембранно-электродных блоков генерации водорода.

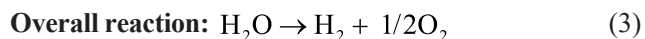
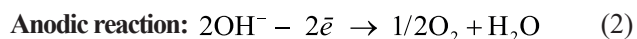
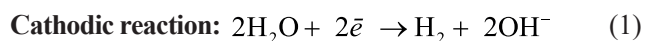
Выводы. Результаты ресурсных испытаний и рабочие характеристики сконструированных вариантов макетов мембранно-электродных блоков с наноструктурированными электродами на основе восстановленного оксида графена, нивелирующие наводораживание и деградацию функциональных материалов, демонстрируют перспективность конструирования генераторов водорода с высокой энергоэффективностью.

Ключевые слова: генерация водорода, наводораживание металлов, мембранно-электродные блоки, нанокompозитные электроды, восстановленный оксид графена, энергоэффективность

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INTRODUCTION

The development of new efficient energy systems using the latest achievements of nano- and chemical technologies, as well as hydrogen energy, is an important objective for energy saving [1–5]. The most promising trend in the design of autonomous energy sources is the creation of integrated energy systems of electrolysis cells with chemical energy converters [3–6]. During the operation of the electrolysis cell, hydrogen and oxygen are formed at the cathode and anode, respectively:



In order to convert the chemical energy of fuel into electricity in autonomous power sources, high-purity (more than 99.95%) hydrogen obtained in alkaline water electrolyzers (AWEs) [2, 7–11] must be used. To improve the energy performance of membrane electrode assemblies (MEAs), as a key component of AWEs, nanostructured functional materials are used [4–6, 12–17]. The MEA design is based on metal gas diffusion electrodes, anode and cathode catalytic layers separated by a polymer membrane (diaphragm). Industrial electrolysis plants have a high level of energy consumption, due to high overvoltages of the hydrogen evolution reaction. This factor contributes to the intense degradation of metal electrodes and electrocatalysts [1–3]. In order to ensure an increased service life of electrolyzers, methods need to be developed to stabilize the functional structural metals for MEAs [4, 18–22].

One of the main causes of the degradation of structural metals in contact with hydrogen in industrial plants can be found in the complex multistep processes of hydrogen dissolution in metals. Hydrogen absorption into metals can cause such phenomena as hydrogen embrittlement of steels, hydrogen hardening, and hydrogen damage during friction [2, 3, 8, 23].

Hydrogen absorption into metals is an integral result of the action of many factors. Its mechanism is mainly determined by the following: the rate of diffusion and transfer of hydrogen in the metal; the localization and concentration of hydrogen in certain regions; the ability of the metal to

interact with hydrogen in places of its localization; and the behavior of the quality parameters of the bulk of the metal (and to a greater extent, the surface layer) which control the ability of materials to adapt to further impacts [23].

Experimental studies into the processes of diffusion, permeability, and solubility of hydrogen in metals confirmed the possibility of penetration and diffusion of hydrogen deep into the material through the crystal lattice. Possessing a high level of energy, hydrogen atoms can be adsorbed on the interface, nonmetallic inclusions, microvoids, and other collectors. In this case, the rate of diffusion is comparable to the rate of crack development.

The mechanisms of hydrogen absorption, as well as the effect of hydrogen embrittlement and hydrogen transfer on the destruction of the crystal lattice of materials and on the changes in physicochemical properties, are studied using various diffusion models. In this case, the contact surface of the materials can be considered, on the one hand, as the surface of a metal catalyst capable of forming hydrides and, on the other hand, of a catalyst which causes the destruction of substances. Analysis of data on these processes is especially necessary when constructing models of degradation of MEAs of hydrogen generators and searching for efficient methods to increase their service life.

In this work, the support in MEAs was porous nickel (PN) formed by the nanotemplate method on an aluminum matrix, and the electrocatalysts were platinum–nickel and palladium–nickel bimetallic nanoparticles (anode and cathode, respectively). The control of flows of gases (hydrogen and oxygen) is one of the most important technological stages of MEA operation. In industrial electrolyzers at high current densities, it is the use of PN with a large active surface area that ensures the efficient removal of gas bubbles to prevent hydrogen absorption into metals for the efficient operation of catalysts [13, 18–22, 24–26].

Various techniques are used [23–29] to increase the degradation resistance of metal electrodes when exposed to hydrogen. The modification of metals with trace amounts of platinum metals, in particular, as well as alloying of metals with copper, aluminum, and calcium, can enhance the resistance of metals to hydrogen absorption. This can also prevent the formation of reactive hydrogen species on the surface of metals, and inhibit corrosion processes by the formation of oxide–hydroxide films. One promising trend to prevent intense hydrogen absorption into metal structures is the possibility of using graphene, which has a high volumetric density of hydrogen storage [28, 29].

This study aimed at assessing the possibility of preventing hydrogen absorption into functional structural metals of nanostructured hydrogen-generating MEAs by using electrocatalytic matrices based on reduced graphene oxide (rGO). The basic criteria for hydrogen absorption into metals were energy efficiency and stability of the hydrogen generation process.

EXPERIMENTAL

The MEA electrodes were produced of platinum–nickel (Pt–Ni) and palladium–nickel (Pd–Ni) bimetallic particles. The precursors for the synthesis of nanoparticles were aqueous solutions of K_2PtCl_4 , $PdCl_2$, and $NiCl_2$ (*Sigma-Aldrich*, USA). PN with a thickness of 2 mm and an average pore diameter of less than 50 μm was used as the main support [22]. The molar ratio of bimetallic particles was 1:1 at a mass loading of palladium at the cathode of $m_s = 0.5 \text{ mg/cm}^2$ and a mass loading of platinum at the anode of 0.8 mg/cm^2 . The sizes of palladium–nickel and platinum–nickel bimetallic nanoparticles ranged from 4 to 9 nm.

In order to estimate the degree of degradation of the electrodes, XC-72 carbon black (*Cabot*, USA) and rGO, obtained according a published procedure [6], were studied as supports.

The energy efficiency and stability of hydrogen generation were assessed using a 600 ETS Electrolyzer Test System (*Scribner Associates Inc.*, USA), consisting of a controller with a potentiostat, a power supply, gas reagent supply systems, and sensors of temperatures, flows, and contents of hydrogen and oxygen. The controller was connected to the electrolyzer by cables for electropositive and electronegative currents (I+, I–), voltage power cables (V+, V–), and auxiliary wires (A+, A–) (Fig. 1). Voltammograms were recorded and the stability of the MEA operation was tested using a two-electrode circuit (Fig. 1).

The MEA was a key part of the electrolysis cell. The MEA consisted of $7 \times 7 \text{ cm}$ PN-based gas diffusion bimetallic electrodes with the ability to control the process temperature from 25 to 80°C , as well as a commercial Sustainion® X37-50 anion exchange membrane (*Fuel Cell Store*, USA) placed between the anode and cathode materials [14]. The stability of the MEA during the electrolysis of water was tested at current densities from 0.05 to 0.6 A/cm^2 for 180 h. The operating voltage was varied in the range from 1.3 to 3 V.

Electrolyzers can use MEAs of two types: standard and zero-gap [19, 21]. The disadvantages of the second type of MEA include the need

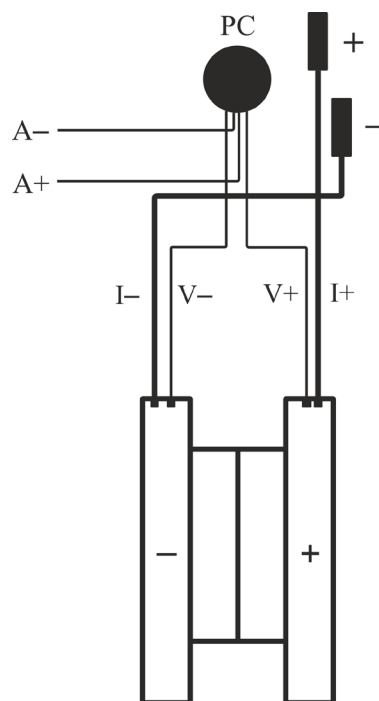


Fig. 1. Two-electrode cell connection diagram: (+), positive electrode (anode); (–), negative electrode (cathode); A, auxiliary electrode; and PC, power cable.

to remove hydrogen and oxygen from the electrodes. In this work, the standard variant (Fig. 2) of the design was chosen due to its ease of assembly and low degradation of the electrolyte. The removal of gaseous reaction products was facilitated by using electrode matrices based on PN with a developed pore system and carbon materials in the gas diffusion layer.

RESULTS AND DISCUSSION

This work studied the effect of the localization of electrocatalysts on the process of hydrogen absorption into structural functional materials of MEAs during hydrogen generation. Three variants of localization of bimetallic nanocatalysts in the MEA during cathodic hydrogen evolution were considered. The three variants differ in the type of support matrix, on which the electrocatalysts are located, i.e., in the localization of hydrogen release and the place of contact with the structural material. The following support matrices were used: PN, an electrode material; carbon black, a standard component of the gas diffusion layer; and rGO, the first proposed component of the gas diffusion layer for studying the hydrogen absorption process.

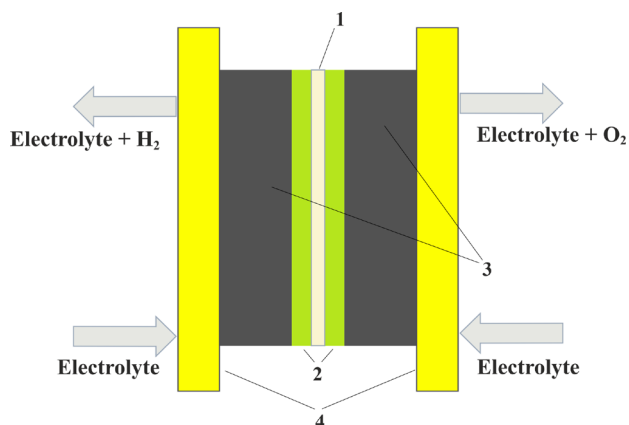


Fig. 2. Scheme of membrane electrode assembly (MEA): (1) anion exchange membrane, (2) gas diffusion layers, (3) electrodes based on porous nickel (PN), and (4) bipolar plates.

Variant 1: the support matrix is PN; Pd–Ni bimetallic nanocatalyst with a mass loading of metals of $m_s = 0.5 \text{ mg/cm}^2$.

Variant 2: the support matrix is XC-72 carbon black; Pd–Ni bimetallic nanocatalyst with a mass loading of metals of $m_s = 0.5 \text{ mg/cm}^2$.

Variant 3: the support matrix is rGO; Pd–Ni bimetallic nanocatalyst with a mass loading of metals of $m_s = 0.5 \text{ mg/cm}^2$.

A characteristic of the efficient operation of the MEA is the specific energy consumption W ($\text{kW}\cdot\text{h/m}^3 \text{ H}_2$) of the AWE process, determined by the ratio of the power P (kW) of the electrolysis process to a unit volume of generated hydrogen:

$$P = I \times U_n. \quad (4)$$

Herein, I is the current in the MEA, A; and U_n is the rated voltage of the electrolysis process, V.

When an electric current passes through the MEA, the energy consumption potential of the electrodes shifts due to additional energy consumption to compensate for the slow transfer of electrical charges and the destruction of structural metals in the process of hydrogen generation. With increasing current density, the voltage increases, and so does the energy consumption for electrolysis. The change in the electrode potential caused by the flow of electric current through the system relative to its value in the absence of current is determined by overvoltage:

$$\eta = U_j - U_0, \quad (5)$$

wherein U_j and U_0 are the voltages in the presence and absence of electric current, respectively. The hydrogen evolution overvoltage η can be calculated by the Tafel equation

$$\eta = b \cdot (\lg j - \lg j_0), \quad (6)$$

wherein j is the operating current density, A/cm^2 ; j_0 is the exchange current density, A/cm^2 ; b is the temperature-dependent constant, V.

Figure 3 presents the experimental dependences of the hydrogen overvoltage on the current density for three variants of MEA with different localization of hydrogen evolution using electrodes based on rGO. It also shows comparison with samples based on carbon black and PN at a temperature of $t = 80^\circ\text{C}$ in a 7M KOH electrolyte. The results demonstrating a decrease in the overvoltage of cathodic hydrogen evolution are typically interpreted as an increase in the exchange current density j_0 (see Eq. (6)).

In order to assess the stability of hydrogen generation, the above three variants of MEA were tested for 180 h, temperatures of 60 and 80°C , and current densities up to 600 mA/cm^2 . Figure 4 shows the time dependence of the operating voltage in three variants of MEA at a temperature of 80°C and a current density of 600 mA/cm^2 .

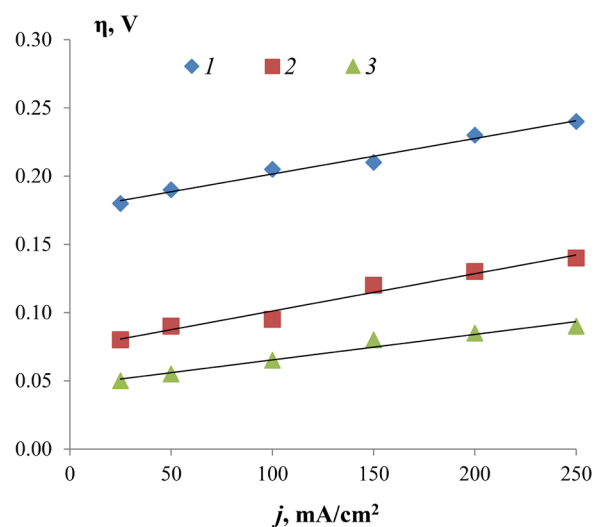


Fig. 3. Polarization curves of cathodic hydrogen evolution for three variants of MEA: (1) PN, (2) XC-72 carbon black, and (3) reduced graphene oxide (rGO).

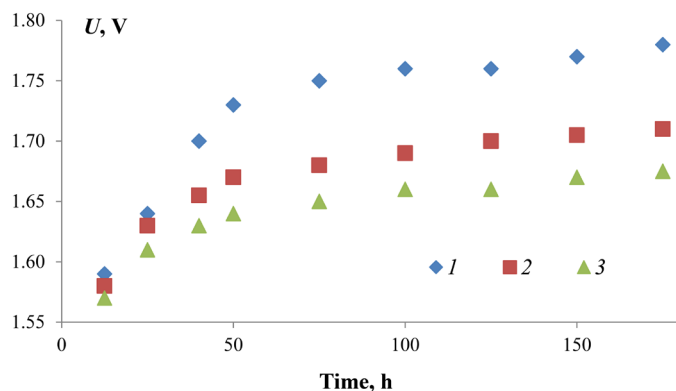


Fig. 4. Dependence of the voltage in three variants of MEA on the operating time of the alkaline water electrolyzer (AWE) at a current density of 600 mA/cm² and a temperature of 80°C: (1) PN, (2) XC-72 carbon black, and (3) rGO.

At the first stage of service life tests (up to 15–20 h), the voltages in all MEAs corresponded to the same content of the Pd–Ni electrocatalyst and differed insignificantly. However, at the next stage of hydrogen generation, the voltage in the MEA with the support matrix based on PN (variant 1) increased significantly in comparison with the carbon variants of support matrices (variants 2 and 3). At the same time, after 15 h of testing, the minimum voltage was observed for variant 3 of MEA with the rGO-based support matrix. This trend continued throughout the service life tests.

The main disadvantages of industrial hydrogen generators are: firstly, high specific energy consumption (more than 4 kWh for generation of 1 m³ H₂ with a long service life and elevated temperatures); and secondly, an increased content

of platinum metals (more than 1–2 mg/cm²). Table 1 presents the specific energy consumption in three variants of nanocomposite MEAs for hydrogen generation at current densities of 500 and 600 mA/cm² and temperatures of 60 and 80°C.

The table provides specific energy consumption data for three variants of nanocomposite MEAs at current densities of 500 and 600 mA/cm², temperatures of 60 and 80°C, and a service life of up to 180 h. These results correspond to the process of generation of high-purity hydrogen with a maximum hydrogen productivity of more than 14 · 10⁻³ m³/h. At a hydrogen generation temperature above 90°C and a current density above 700 mA/cm², intense agglomeration of Pt–Ni and Pd–Ni bimetallic nanoparticles was observed in the first 30–40 h of the service life tests. The energy efficiency of hydrogen-generating MEAs obtained with carbon nanocomposite electrodes exceeds the characteristics of commercial hydrogen generation electrolyzers and is on a par with promising modern pilot plants [4, 6, 12, 13].

The rGO-based variant of MEA demonstrated the best results in energy efficiency and stability of hydrogen generation, especially in comparison with the PN-based one. However, the question arises about the causes of this phenomenon and the effect of the structure of the support matrix on increasing the rate of hydrogen formation in the cathodic evolution reaction. This reaction is a complex multistep process and is necessary to resolve problems of energy saving and create anti-corrosion coatings. For most metals, the reaction rate of hydrogen formation is determined by the rate of discharge of hydrogen ions. The removal of adsorbed hydrogen occurs by the Heyrovsky reaction, i.e., the mechanism of electrochemical desorption:

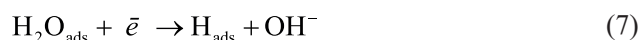
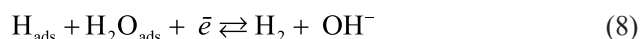
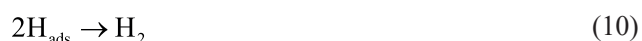
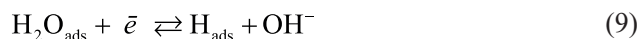


Table. Specific energy consumption of electrolysis cells per 1 m³ of H₂ (pressure 1 bar) at different current densities

<i>t</i> , °C	Specific energy consumption per 1 m ³ of H ₂ , kWh·m ³ of H ₂					
	<i>j</i> = 500 mA/cm ²			<i>j</i> = 600 mA/cm ²		
	1	2	3	1	2	3
80	4.01	4.05	4.10	4.06	4.08	4.13
60	3.98	4.18	4.24	4.14	4.21	4.28



The dependence of the binding energy of adsorbed hydrogen on the metal surface on the overvoltage at the electrode suggests that cathodic hydrogen evolution can also occur through other mechanisms:



Thus, the electrochemical desorption reaction can also be the rate-limiting step of the process.

On platinum catalysts at low overpotentials, the rate-limiting step of the process is the recombination of hydrogen atoms [27]. Due to the significant heterogeneity of the electrode surface, the reaction can occur by various mechanisms. This can have a significant impact on the shape of the polarization curves. This confirms the presence of excess surface hydrogen concentration on the polarization side, caused by the rate-limiting step of hydrogen removal and hydrogen penetration through the metal.

When analyzing the causes of the decrease in the energy efficiency and stability of hydrogen generation, the hydrogen saturation of structural materials of MEA needs to be taken into account. The hydrogen saturation of metals occurs by the dissociation on the surface of the support. In similar cases, the saturation of metals is caused by the penetration of hydrogen atoms into them as a result of corrosion reactions, or during cathodic polarization. Metals lose their physicochemical properties, causing degradation of the functional materials of the MEA. Measures are needed to prevent the formation of reactive hydrogen species and ensure the efficient removal of the generated hydrogen from electrocatalysts. In this work, rGO was chosen

as the material for preventing these degradation processes. Figure 5 presents the model of the structure of unreduced graphene oxide. Figure 6 shows a micrograph of the rGO support matrix with Pd–Ni nanoparticles.

Hydrogen generation involves the reduction of the initial graphene oxide to rGO, preventing the formation of reactive hydrogen species (atoms, radicals) and hydrogen absorption into the active surface of metals (see Eq. (7)). The efficiency of the removal of the generated hydrogen from electrocatalysts and nickel electrodes is ensured by two factors: the high volumetric density of hydrogen storage in both the layered structure of rGO (Fig. 6); and the porous structure of individual graphene layers. Moreover, the hydrogen production by the alkaline electrolysis of aqueous solutions is carried out at elevated temperatures (60–80°C). This leads to hydrogen desorption by thermal fluctuation vibrations and hydrodynamic deformation of graphene layers [28, 29]. Intensification of the removal of molecular hydrogen from the zone of the electrocatalytic process increases the rate of the rate-limiting stage of recombination of adsorbed hydrogen atoms on platinum group metals (platinum, palladium) [27] and reduces the hydrogen absorption of metallic structural materials of MEA by reactive hydrogen species.

CONCLUSIONS

The research studies the possibility of preventing hydrogen absorption into functional structural materials of nanostructured hydrogen-generating MEAs. The criteria for process of hydrogen absorption of metals were energy efficiency and stability of the hydrogen generation process. For the first time, the results of service life tests using electrocatalytic matrices based on rGO demonstrated the stability and high electrocatalytic activity of functional nanocomposite electrodes at elevated temperatures (up to 80°C) and current densities (more than 600 mA/cm²). The specific

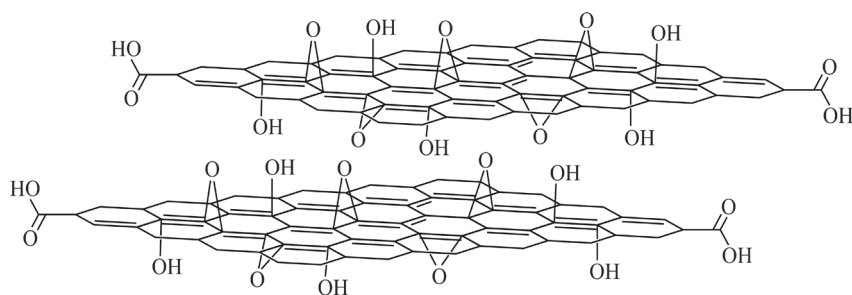


Fig. 5. Model of graphene oxide.

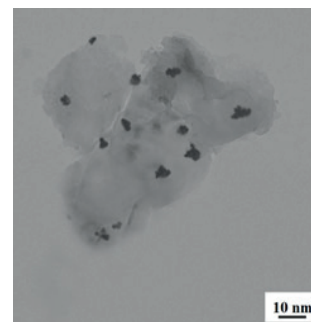


Fig. 6. Micrograph of the rGO support matrix with Pd–Ni nanoparticles.

energy consumption and performance characteristics of the developed prototypes of AWEs with nanocomposite electrodes based on rGO, preventing the hydrogen absorption and degradation of functional materials, indicate that it shows the potential to create hydrogen generators with a high level of energy efficiency.

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Authors' contributions

M.V. Lebedeva – planning and conducting experiments, drawing up methods;

A.V. Ragutkin – formulation of the scientific concept, goals and objectives of the work, processing and interpretation of experimental results;

I.M. Sidorov – analysis of experimental data;

N.A. Yashtulov – writing the manuscript and leading a scientific group.

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No author has a financial or property interest in any material or method mentioned.

The authors declare no conflicts of interest.

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