A gas sensor based on ZnO has been designed, which demonstrates sensitivity to NH_3 under standard conditions (temperature, 25 °C; pressure, 101.3 kPa). The experimental sample was manufactured by magnetron sputtering at direct current. A VUP-5M vacuum unit with an original material-saving magnetron was used to produce ZnO films. To analyze the efficiency of the gas sensor to ammonia (NH_3) under standard conditions, its operating characteristics were studied. The concentration of NH_3 for investigating operating characteristics was chosen at the level of 25 ppm. To determine the resistivity of the contacts of the instrument structure, the current-voltage characteristics of the gas sensor were examined in the voltage range between –100 and +100 V. Based on the results of investigating the current-voltage characteristics, which have a linear character, the resistivity of the contacts was confirmed. To study the sensitivity of the gas sensor to the target gas, the change in resistance of the sensitive layer of the gas sensor under the influence of NH_3 with a concentration of 25 ppm under standard conditions was explored. The study results demonstrated the high sensitivity of the gas sensor to the target gas – at the level of 229 relative units. The investigation of the response and recovery time of the gas sensor showed that the ZnO-based gas sensor has a response and recovery time of 20 and 26 s, respectively. The selectivity of the ZnO-based gas sensor was studied. The selectivity study was carried out by determining the sensitivity of the gas sensor in the presence of vapors of various gases, namely methanol, ethanol, acetone. The study results showed that the reaction to ammonia is selective compared to the reaction to other gases. The results of examining the working characteristics of the ammonia gas sensor demonstrate the high efficiency of its application under standard conditions and a low concentration of the target gas

Keywords: ZnO, gas sensor, magnetron sputtering, standard temperature, ammonia

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DESIGN OF AMMONIA SENSOR BASED ON ZnO FOR ANALYZING HAZARDS AT CRITICAL INFRASTRUCTURE

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1. Introduction

Ammonia (NH_3) is of great commercial interest due to its unique properties for use as a nitrogen source for soil fertilizers, a neutralizer in the oil industry, a gas refrigerant in industrial systems, and in the pharmaceutical industry [1]. The world produces about 200 million tons of ammonia per year. The scenario of sustainable development of the International Energy Agency predicts that by 2050 the demand for ammonia will grow at least twice [2]. Accordingly, the number of hazardous substances at critical infrastructure facilities will also increase. NH_3 is a colorless flammable gas with the smell of ammonia, a colorless liquid at a temperature of –33.4 °C, which solidifies at a temperature of –78 °C. According to the globally agreed system of classification and labeling of chemical substances, NH_3 represents three classes of hazards at once: physical, health, and environmental hazards [3]. In an open space, ammonia-air mixtures do not explode in the detonation mode, but in closed volumes (rooms, equipment) an explosion is possible. NH_3 is dangerous by inhalation of vapors, contact with skin and mucous membranes. In the atmosphere, the normal level of NH_3 is within (1–5 ppb). Short-term human exposure for 15 min should be limited to a concentration of 25–35 ppm, and the time-weighted average concentration over an 8-hour period should not exceed 25 ppm [4]. Prolonged exposure to dangerous levels of NH_3 can cause lung disease in both humans and animals. Regarding the impact on the environment, it should be noted that NH_3 is considered an environmental pollutant because it is highly active and forms aerosols such as ammonium nitrate and ammonium sulfate [5]. As a result, these nano-sized NH_3 aerosols create smog, which exhibits a temperature-lowering effect and, as a result, negatively affects the global greenhouse balance [2]. Therefore, it is extremely important to monitor and control the concentration of NH_3 at critical infrastructure facilities, both from the point of view of explosion and fire hazards [6] and to ensure the safety of workers and environmental protection [7]. The research of new inexpensive and effective sensors for monitoring and controlling the concentration of ammonia in the atmosphere is the need of the hour. The main performance characteristics of the sensor include sensitivity, selectivity, response and recovery time, stability upon repeated exposure to the target gas, ease of manufacturing, and low energy consumption [8]. Most of the commercially available gas sensors have high sensitivity only at elevated operating temperatures, therefore the instrument structures are equipped with microheaters [9]. But frequent switching between heating and normal atmospheric conditions leads to structural transformation of the sensitive layer, phase separation, grain growth of the sensitive element. Elevated operating temperatures also lead to deterioration of the contacts and performance of the heater, which, in turn, leads to low stability and service life of the sensor [10]. Therefore, the detection of NH_3 at room temperature would be one of the best solutions to solve the problem of sensor degradation during operation. The results of such research are needed in practice to build inexpensive and effective devices, using a method suitable for large-scale production.

2. Literature review and problem statement

The most common methods for detecting NH_3 can be classified into three main categories. The first category includes optical sensors using adjustable diode laser spectroscopy, electrochemical sensors, surface acoustic wave sensors. The second one – sensors on field effect transistors. The third includes solid-state sensors based on metal oxide.

Gas sensors that use optical absorption are able to overcome obstacles that other gas sensors that use contact measurement methods usually have, such as measurement error due to the long-term memory effect [11]. Such sensors are easy to operate, may have high selectivity, high speed, and have degradation resistance [12]. However, the performance of the optical sensor is affected by external light. Electrochemical sensors have low energy consumption, portability, high sensitivity, and selectivity [13]. Among the disadvantages of electrochemical sensors worth noting are the high cost and low degradation resistance. Metal oxide-based sensors have attracted considerable attention in the field of gas measurement due to advantages such as simplicity, low cost, and flexibility in fabrication, as well as good process compatibility. Metal oxide-based gas sensors, especially SnO_2, ZnO, WO_3, TiO_2, and MoO_3, are the most widely used metal oxides for NH_3 detection. The new carbon nanotube-based gas ionization sensor described in [14] demonstrates high accuracy, repeatability, and stability. W_xO_y nanowires are very sensitive to ammonia levels below ppm and ppb at room temperature, which is explained by their small diameter, large surface area, and non-stoichiometric crystal structure [15, 16]. High sensitivity and selectivity of MoO_3 films to the target gas NH_3 was found in [17]. However, resistance was found to slowly increase over time. These changes can be caused by diffusion processes. Basically, as a gas-sensitive material, metal oxides are divided into *n*-type and *p*-type semiconductor metal oxides.

The authors of paper [18] designed a gas sensor based on a Schottky diode, obtained as a result of contact between ZnO nanorods and Au nanoparticles, aimed to detect gaseous ammonia at room temperature with a power consumption of 625 μW. Due to this contact, a 6-fold increase in sensitivity to NH_3 (40 ppm) compared to other gases was achieved. The Schottky diode provided better selectivity but had a long response/recovery time (28 min/78 min), which calls into question its practical application. In [19], the authors designed a highly selective NH_3 gas sensor based on tin-titanium dioxide/reduced graphene/carbon nanotube ($Sn-TiO_2 \cong rGO/CNT$) nanocomposites. The $Sn-TiO_2 \cong rGO/CNT$ nanocomposite gas sensor demonstrated ultrahigh selectivity to NH_3 (250 ppm) against toluene, dimethylformamide, acetone, ethanol, methanol, isopropanol, formaldehyde, hydrogen, carbon dioxide, acetylene at room temperature. Response/recovery time (96 s/66 s) at a concentration (250 ppm) of NH_3. However, a concentration of 250 ppm NH_3 can be fatal. The operating parameters of the proposed instrument structure at concentrations of the target gas ten times lower are unknown. The authors of [20] designed a cheap solid-state gas sensor based on paper electronics for the selective detection of NH_3 gas with a detection capability above 1 ppm. The sensor uses perovskite halide $CH_3NH_3PbI_3$ as the active sensor material grown on paper. This paper sensor works at room temperature. The current through the paper sensor increases by an order of magnitude when exposed to as little as 10 ppm NH_3 gas. Response/recovery time is ~135 s/112 s and estimated resolution is ~10 ppm. However, the proposed gas sensor has low degradation resistance. Several other room temperature NH_3 gas sensors have been designed using nanocomposites. In [21], the NH_3 gas sensor was obtained on the basis of a polythiophene/molybdenum oxide nanocomposite. In [22], an NH_3 gas sensor was obtained using an organic PEDOT:PSS layer. A flexible NH_3 gas sensor was proposed by the authors of paper [23]. However, they all have low degradation resistance.

Thus, further research is needed to design NH_3 gas sensors that have high sensitivity, high speed, degradation resistance, and low cost. ZnO, an n-type semiconductor with a direct bandgap at room temperature, has high electron mobility and sensitivity to many gases [24]. In addition, ZnO has oxygen molecules adsorbed on its surface, which can form ionized forms capable of capturing electrons from the conductive zone of the oxide, creating a depleted layer on the oxide surface [25, 26]. These properties encourage the use of ZnO in the development of gas sensors sensitive to low concentrations of NH_3. Another advantage regarding the research of gas sensors based on ZnO is that there are different ways of obtaining a sensitive layer, and accordingly, different morphologies. ZnO morphologies are known as 0D (quantum dots – QDs), 1D (nanorods, nanoribbons, nanotubes, nanofibers, and nanowires), 2D (nanosheets and nanofilms) and 3D (nanoflowers, porous spheres, and hollow spheres) nanomaterials. Both the morphology and the structure of the sensitive layer of the ZnO-based gas sensor affect its initial operating parameters.

Thus, an unsolved problem is the mandatory heating to high temperatures of the sensitive layer of the ZnO-based gas sensor to obtain a response to the target gas, which reduces its degradation resistance. Obtaining a ZnO-based gas sensor capable of detecting the presence of a target gas at room temperature requires the development of a technology for designing such an instrument structure.

3. The aim and objectives of the study

The aim of our work is to design a gas sensor based on ZnO to the target gas NH_3 under standard conditions. This will make it possible to increase the degradation resistance of the instrument structure and, accordingly, its service life.

To achieve the goal, the following tasks must be solved:

– to investigate the current-current characteristics of a ZnO-based gas sensor obtained by magnetron sputtering at direct current;

– to investigate the operating characteristics of a gas sensor based on ZnO obtained by magnetron sputtering at direct current under the influence of a target gas under standard conditions.

4. Research materials and methods

4. 1. Obtaining the tested samples of the ZnO-based gas sensor

Obtaining the studied samples of the gas sensor based on ZnO was carried out by the method of direct current magnetron sputtering. A VUP-5M vacuum unit was used to obtain the films [27].

The target for obtaining ZnO on the surface of the substrate was a zinc target (99.99 % pure). The length of the discharge gap, which is the distance between the magnetron and the substrate, was 70 mm. The power of the magnetron was 0.2 W/cm^2. High-purity argon was used as an inert gas, and oxygen as an active gas. The outlet pressure in the vacuum chamber was 3×10^{-5} mm Hg, the working pressure of the argon-air mixture during the spraying process is $(2.1–2.6)\times10^{-2}$ mmHg. The substrate temperature was 300 °C. The deposition rate was 12 Å/s [28]. A helium-neon laser with a wavelength of 633 nm was used to determine the thickness of the films. The resulting films had a thickness of 2–2.5 μm.

For the manufacture of a gas sensor, contacts were applied to the obtained samples, which were created by vacuum deposition of Al films with a thickness of 300 nm at a residual gas pressure of 10^{-4} Pa through a special mask. The connection of aluminum contacts with the wires of the outer circuit was carried out by using conductive glue based on silver «Kontaktol». Fig. 1 shows the investigated gas sensor based on ZnO.

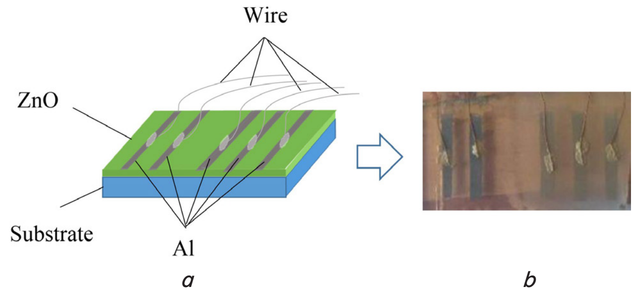

Fig. 1. The studied gas sensor based on ZnO: *a* — schematic representation of the device structure; *b* — a photograph of the instrument structure

The distance between two adjacent aluminum strips is 3 mm, the contact length L is 15 mm.

4. 2. Determining the concentration of the target gas NH_3 for the study of the performance characteristics of the ZnO-based gas sensor

NH_3 represents three classes of hazards at once: physical, health hazard, and environmental hazard, so it is necessary to determine the lowest concentration of the target gas to analyze the efficiency of the gas sensor.

In terms of human risk, the time-weighted average concentration over an 8-hour period should not exceed 25 ppm.

From the point of view of fire and explosion hazard, the mass lower concentration limit of flame spread was determined. According to [9], the lower concentration limit of flame propagation is 16.2 %. Using expression (1), the volume concentration of NH_3 was converted into mass:

$$\varphi' = \varphi\frac{10\mu}{V_\mu}, \tag{1}$$

where φ is the lower concentration limit of flame propagation, μ is the molar mass, and V_μ is the molar volume under standard conditions. It was found that an explosive concentration of ammonia is formed at a value of $159\cdot10^3$ ppm.

Thus, the minimum ammonia concentration at which a ZnO-based gas sensor must be tested is 25 ppm.

4. 3. Study of volt-ampere characteristics of a gas sensor based on ZnO

The ammeter-voltmeter method was used to study the current-voltage characteristics (CVC) of the gas sensor with the help of the installation, the scheme of which is shown in Fig. 2.

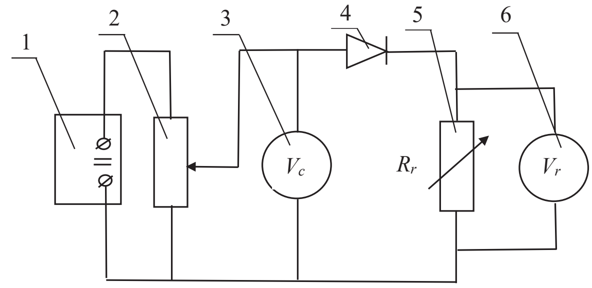

Fig. 2. Diagram of the installation for measuring volt-ampere characteristics under static mode: 1 — source of stabilized voltage TES-5050; 2 — resistive voltage divider; 3 — voltmeter U7-37; 4 — the investigated gas sensor; 5 — cartridge of reference resistances R-33; 6 — voltmeter U7-38

The stabilized voltage source TES-5050 provides a specified voltage drop V_c at the terminals of the measuring circuit, controlled by a digital voltmeter U7-37.

The values of forward I_f and reverse I_r currents of the sample are determined by measuring the voltage drop V_r on the reference resistor R_r with a U7-38 digital voltmeter, after which the current is calculated according to the formula:

$$I = V_r / R_r. \quad (2)$$

The voltage drop across sample V is calculated using the following formula:

$$V = V_c - V_r. \quad (3)$$

The study of the voltage-ampere characteristics of the gas sensor is necessary to determine the ohmicity of the contacts of the instrument structure.

4. 4. Studying the operating characteristics of a ZnO-based gas sensor under the influence of NH_3

Performance characteristics of a gas sensor include sensitivity, response time, relaxation time, selectivity, and degradation resistance.

The study of the operating characteristics of the gas sensor based on ZnO was carried out using the installation, the scheme of which is shown in Fig. 3.

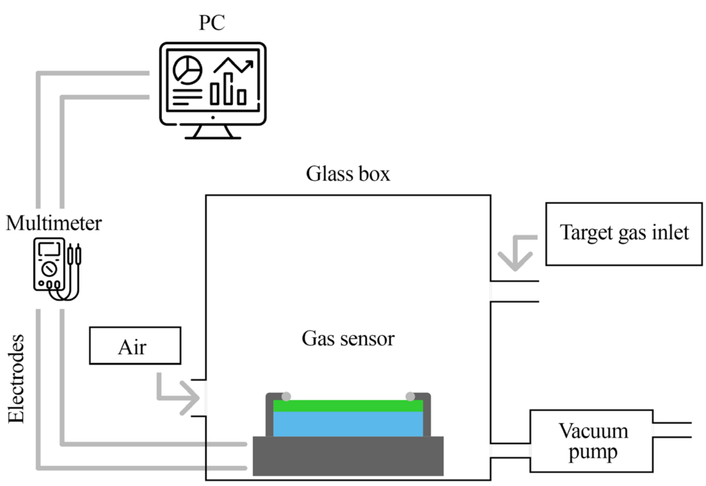

Fig. 3. Diagram of the installation for the study of the operating parameters of the gas sensor

The studied sample of the gas sensor based on ZnO was placed in a glass box with a volume of 3.6×10^{-2} m^3. (NH_3) was supplied as the target gas. The glass box was filled with the studied gas using an air pump. The target gas concentration was controlled by flow rate and delivery time at ambient temperature. All sensitivity characteristics were studied at room temperature. The resistance was continuously monitored while a specific level of (NH_3) concentration (ppm) was fed into the glass box. After reaching a stationary response, a change in the resistance of the sample relative to the initial value was noted. The value of sensitivity (S) was calculated as:

$$S = \frac{R_0}{R_g} 100\,\%, \quad (4)$$

where R_0 is the resistance of the gas sensor in air, R_g is the resistance of the gas sensor in the atmosphere of the target gas.

Response time and recovery were calculated from a time versus resistance plot, defined as the time required to achieve 90 % and 10 % change in resistance from the initial value, respectively.

5. Results of investigating the designed ZnO-based NH_3 gas sensor under standard conditions

5. 1. Results of studying the volt-ampere characteristics of the ZnO-based gas sensor

The results of our study of the volt-ampere characteristics of the ZnO-based gas sensor are shown in Fig. 4.

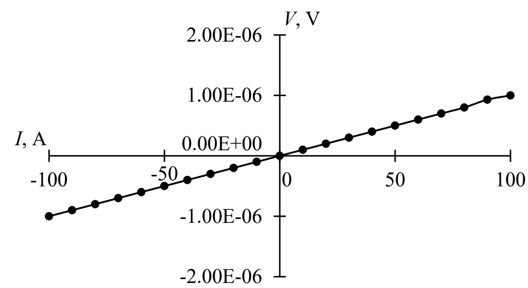

Fig. 4. The volt-ampere characteristic of a ZnO-based gas sensor

As can be seen from Fig. 4, the volt-ampere characteristic of the ZnO-based gas sensor is linear.

5. 2. Results of investigating the operating parameters of the ZnO-based gas sensor under the influence of NH_3

The results of our study of the sensitivity of the reaction time and the relaxation time of the gas sensor based on ZnO under the influence of the target gas NH_3 with a concentration of 25 ppm are shown in Fig. 5.

The results of the gas sensor selectivity study are shown in Fig. 6.

Fig. 7 shows the results of a study of the stability of the gas sensor for 10 days.

Degradation resistance was determined by repeated exposure to NH_3 at a concentration of 25 ppm for 10 days.

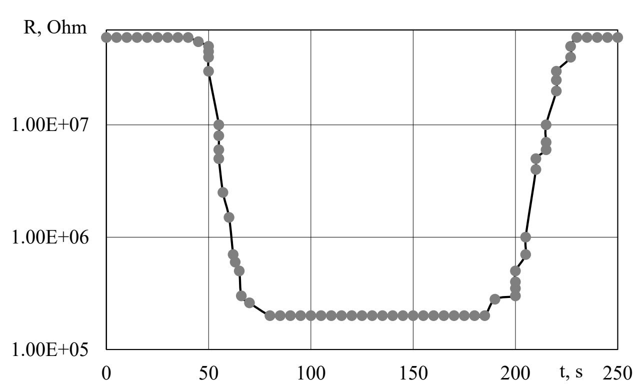

Fig. 5. Resistance change of the gas sensor based on ZnO under the influence of NH_3 with a concentration of 25 ppm

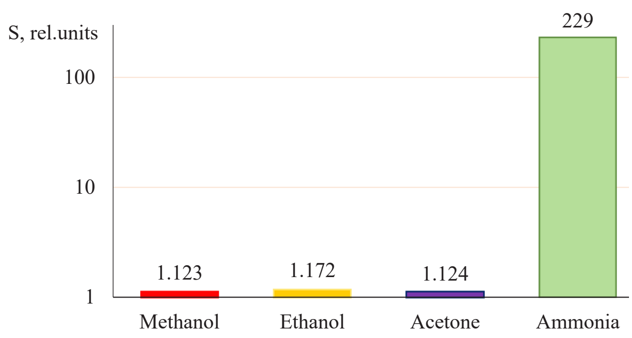

Fig. 6. Selectivity of a ZnO-based gas sensor under the influence of gases with a concentration of 25 ppm

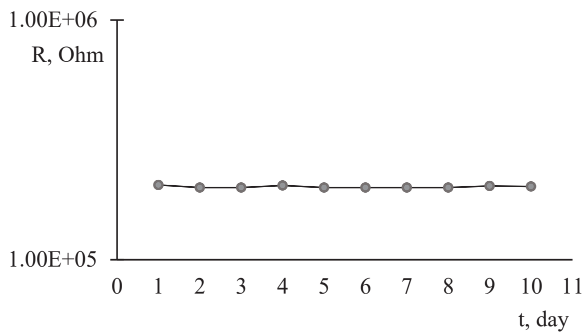

Fig. 7. Degradation resistance of a ZnO-based gas sensor

6. Discussion of results of the ZnO-based gas sensor research

In the work, a gas sensor based on ZnO obtained by the method of direct current magnetron sputtering was investigated. This method of forming the instrument structure was chosen among others due to the following advantages. The DC magnetron sputtering method has a high deposition rate at low values of the working gas pressure. In this method, there is no overheating of the substrate, a low degree of contamination of the obtained films, the possibility of obtaining samples of uniform thickness on a large area of the substrate. To confirm the ohmic nature of the contacts to the gas sensor based on ZnO, studies of the volt-ampere characteristics of the instrument structure were conducted at room temperature in the voltage range between –100 and +100 V. According to the result of the study of the volt-ampere characteristics (Fig. 4), which has a linear character, ohmic contacts were confirmed. The study of change in the resistance of the sensitive layer of the gas sensor under the influence of the target gas NH_3 with a concentration of 25 ppm at standard temperatures showed a high sensitivity at the level of 229 rel. units. The gas sensor demonstrated a fast response and recovery time of 20 and 26 s, respectively. One of the important operating parameters of a gas sensor is selectivity. High sensitivity to the target gas but low selectivity limits the possibilities of independent use of the gas sensor, which is typical for gas sensors based on metal oxides. Thus, the study of the selectivity of the gas sensor was checked in the presence of vapors of various gases, namely methanol, ethanol, acetone. The results of the study showed that the reaction to ammonia is selective compared to the reaction to other gases (Fig. 6). This value of the reaction to ammonia compared to other gases is due to the strengthening of the catalytic effect on the ZnO surface. This, in turn, increases the concentration of negatively charged oxygen ions on the surface of the gas sensor. Upon introduction of a reducing gas such as ammonia vapor, the vapor reacts with adsorbed oxygen on the surface, resulting in the release of trapped electrons onto the surface.

Analysis of the degradation resistance of the gas sensor under the influence of the target gas showed the stability of the device structure during repeated exposure for 10 days.

In contrast to the results reported in [8, 9], the ammonia gas sensor demonstrates a high efficiency of detecting the target gas under standard conditions (operating temperature 25 °C) and a low concentration of 25 ppm. In [8], a gas sensor based on ZnO demonstrates sensitivity to the target gas ethanol only when the substrate is heated to a temperature above 200 °C, which negatively affects its degradation resistance. In [9], a gas sensor based on ZnO demonstrates sensitivity to NH_3 of the target gas only at its concentration of 150 ppm.

The main limitation of this study is that the results can be used only for the detection of reducing gases. The development of gas sensors based on ZnO, which have a high efficiency of detecting redox gases of low concentration at standard temperature, requires further technological solutions.

The lack of analysis of the influence of atmospheric air humidity on the working characteristics of the ammonia gas sensor is the shortcomings of this study. This shortcoming can be eliminated in further studies aimed at establishing the dependence of change in the operating characteristics of the ammonia gas sensor on the level of humidity of the atmospheric air.

7. Conclusions

1. A gas sensor based on ZnO has been designed, which shows sensitivity to NH_3 under standard conditions. The volt-ampere characteristics of the resulting ZnO-based gas sensor showed a linear character, indicating the resistivity of the electrical contacts to the sensitive layer of the device structure.

2. The operating parameters of the gas sensor based on ZnO obtained by the method of magnetron sputtering at constant current under standard conditions were studied. The gas sensor was found to exhibit high sensitivity to a target ammonia gas concentration of 25 ppm at 229 rel. units. The speed of reaction to the target gas and the speed of recovery are 20 and 26 s, respectively. The gas sensor demonstrates high selectivity and degradation resistance.

Conflicts of interest

The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study and the results reported in this paper.

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Data availability

The data will be provided upon reasonable request.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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