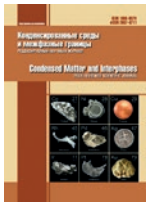

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Structures for photocatalysis based on ZnO with Ag nanoparticles

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Abstract

Purpose: This paper aims to establish the regularities of the deposited silver influence on the catalytic activity of zinc oxide. Silver nanoparticles make a promising component for improving the catalytic performance of semiconductor materials through the effect of plasmonics.

Experimental: The experimental part included synthesis of specimens with different silver content from 0.2 to 2 wt %. SEM images and AFM scans of the powders were obtained to characterize the specimen surface. EDX spectra and elemental mapping were obtained to analyze the composition. As a result, the uniform deposition of silver on the surface of zinc oxide and the agreement of the estimated composition with the obtained were confirmed. Catalyst activity was evaluated by the degree of degradation of the organic dye Rhodamine 6G. The effect of deposited silver on ZnO surface was analyzed.

Conclusions: The deposition of 0.2 wt % silver increases the activity by 58 %, while addition of 2 wt % leads to an increase in activity by 92 %. According to the data obtained, a positive effect of deposited silver on the photocatalytic activity of zinc oxide was found. Dependence of activity change on the amount of silver reaches saturation when 2 wt % of silver is reached.

Keywords: Zinc oxide, Silver nanoparticles, Photocatalysis, Heterojunction, Ecology

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1. Introduction

Among the global issues humanity is facing, environmental and energy issues are paramount. The consumption of resources and energy, and consequently waste, is increasing at a tremendous rate. Synthetic dyes, antibiotics and plastic microparticles which are toxic substances endangering flora and fauna constitute a significant percentage of the waste discharged. They have a negative effect on aquatic environment reducing the oxygen level, and lead to genetic mutations due to their carcinogenic properties. Therefore, the issue of their neutralization is acute. Various methods to treat wastewater such as biological and physico-chemical treatment are already in use. However, due to the toxicity and complex molecular structure of pollutants, modern and highly efficient treatment methods such as photocatalysis are required.

Due to its ability to operate under green energy conditions, photocatalysis, is one of the key areas in the field of environmental remediation. To date, a significant amount of work has been done on designing and developing photocatalysts for applications such as degradation and transformation of hazardous organic substances, hydrogen generation, and CO_2 and NO_x reduction [1, 2].

Recombination of electron-hole pairs (excitons) serves as a factor reducing photocatalytic activity. An effective method is to create an interface at the boundary of two materials with different forbidden band widths which allows to obtain a heterojunction and spatially separate photogenerated charges due to internal electric field. This leads to an increase in the exciton lifetime [3]. Different types of heterojunctions such as Schottky barrier [4], *p-n* junction (or isotype) heterojunctions [5], van der Waals [6] and faceted heterojunctions [7] are designed and investigated depending on the specific objectives for their application. Each type has its own advantages and disadvantages, so the proper choice of heterojunction is important. Semiconductor materials, especially metal oxides [8], perovskites [9], chalcogenides [10], as well as hierarchical structures including organometallic frameworks (MOFs) [11], covalent organic frameworks (COFs) [12], and MXenes [13] are widely used in modern nanophotocatalysis.

Nanoarchitectonics is a cutting-edge direction in design of new materials including heterostructures. This concept considers various approaches, such as atomic-molecular design, unconventional growth mechanisms, including oriented splicing and mesocrystal formation, use of colloidal quantum dots to extend the optical sensitivity range of porous hierarchical structures, and sol-gel technologies to obtain new nanostructured materials with desired properties [14–16].

Among semiconductor materials, zinc oxide stands out for its strong oxidizing ability, excellent optoelectronic and catalytic properties, high chemical stability, nontoxicity, and low cost [17, 18]. ZnO nanoparticles, which are generally n-type semiconductors, have a forbidden bandwidth of 3.37 eV and many active catalytic centers. However, the photocatalytic activity of the material is limited by its ability to absorb electromagnetic radiation. Properties of this material are strongly dependent on intrinsic point defects structure. This has led to development of various techniques to modify the surface of ZnO specimens [19-20]. For example, it is possible to change the concentration of oxygen vacancies on the surface of the material, to form new adsorption centers, and change the type of conductivity by mechanical activation, electron beam irradiation, or annealing [21, 22].

Modification of ZnO with metallic elements such as Au, Ag and Cu can lead to an improvement in photocatalytic performance of the material due to change in the zone structure and the plasmon effect. Silver nanoparticles are the most interesting among other metal nanoparticles. They have attracted attention because of their unique electrical conductivity, chemical stability, catalytic and antimicrobial activity [23]. Making ZnO-based composites with addition of Ag leads to changes in the properties of the semiconductor material. They act as a modifier of reaction centers and also as a catalyst for decomposition of pollutants [24]. ZnO islands capture photoinduced charge carriers and enhance light absorption, with both effects accelerating redox reactions and enhancing the photocatalytic efficiency of the material [25–27].

Research papers show that deposition and introduction of precious metal nanoparticles are

promising methods to enhance the photocatalytic activity of the material. Thus, in [28], the co-precipitation method was used to synthesize ZnO specimens containing silver. The flaked specimens were finely dispersed. The activity study was carried out using methyl blue. Addition of silver resulted in an increase in activity from 87.7 to 97.7 % when the silver concentration was increased to 1 wt % with all other conditions of the catalyst activity study being equal. The study [29] used a photo-deposition method to produce zinc oxide with silver nanoparticles on the surface. Presence of silver enhanced specimen performance. It was observed that increasing the amount of silver up to 1 wt % leads to a significant increase in photocatalytic activity, but further increase in the amount of silver leads to a decrease in activity. This is explained by blocking of active centers of zinc oxide.

In the course of this study, ZnO-Ag specimens with different silver mass fraction were obtained by chemical reduction of silver nanoparticles. This is a simple method of obtaining zinc oxide-silver composite.

2. Experimental

2.1. Synthesis technique

Commercial powder ZnO all-Union State Standard (GOST) 10262-73, *pure* according to chemical classification, was used as the basis of the composite with the content of the parent substance no less than 99 %, and specific surface of 8-10 m^2/gram.

In order to establish correlation between the composition of ZnO-Ag composite and change in its activity, specimens with different silver mass fraction were synthesized. The composites were obtained by reduction of silver from $AgNO_3$ salt using $NaBH_4$ on ZnO surface in presence of polyvinylpyrrolidone stabilizer (PVP) in accordance with the following procedure:

1) ZnO was dispersed in distilled water on an ultrasonic bath for several minutes;

2) $AgNO_3$ was pre-dissolved in a small amount of distilled water using an ultrasonic bath;

3) the $AgNO_3$ solution was added to ZnO and stirred vigorously for one hour;

4) to prevent active agglomeration of silver during the reduction, PVP solution was added equimolar to $AgNO_3$;

5) the resulting solution was stirred for one hour;

6) $NaBH_4$ solution equimolar to AgNO was used for silver reduction;

As a result, specimens with different ratios of components were obtained.

3. Results and discussion

3.1. Characterization of specimen

The specimens obtained had different coloration ranging from pale yellow to dark gray. Yellow tint can be considered as an indirect sign of silver nanoparticles recovery on the substrate surface. Table 1 shows external characteristics of the specimens obtained.

Using a Bruker scanning electron microscope, SEM images of the surface of the studied specimens were obtained (see Fig. 1). Primary analysis of the images showed that the substrate used was a finely dispersed powder. According to the obtained image of pure zinc oxide at 5000x magnification, it can be noted that the ZnO particles size was in the submicron range. Analysis of modified specimen images (Fig. 1b-d) allows to draw a similar conclusion about the specimen dispersibility.

For further information about the surface of the powder catalyst, the surface was scanned using an atomic force microscope (AFM) Ntegra Prima by *NT-MDT*. Scanning was carried out in semi-contact mode. As a result, surface scans of the ZnO-Ag-2 specimen were obtained (see Fig. 2). Scan analysis allowed to obtain further details about the specimen surface. The powder is comprised of both large formations of more than 1 μm and smaller ones of about 200 nm. The data obtained from these scans do not provide any reliable confirmation of silver nanoparticle deposition on the substrate surface.

Table 1. Characteristics of synthesized samples

Sample	Silver content, wt %	Powder color
ZnO	0	White
ZnO-Ag-0.2	0.2	Light yellow
ZnO-Ag-0.5	0.5	Yellow
ZnO-Ag-2	2	Dark grey

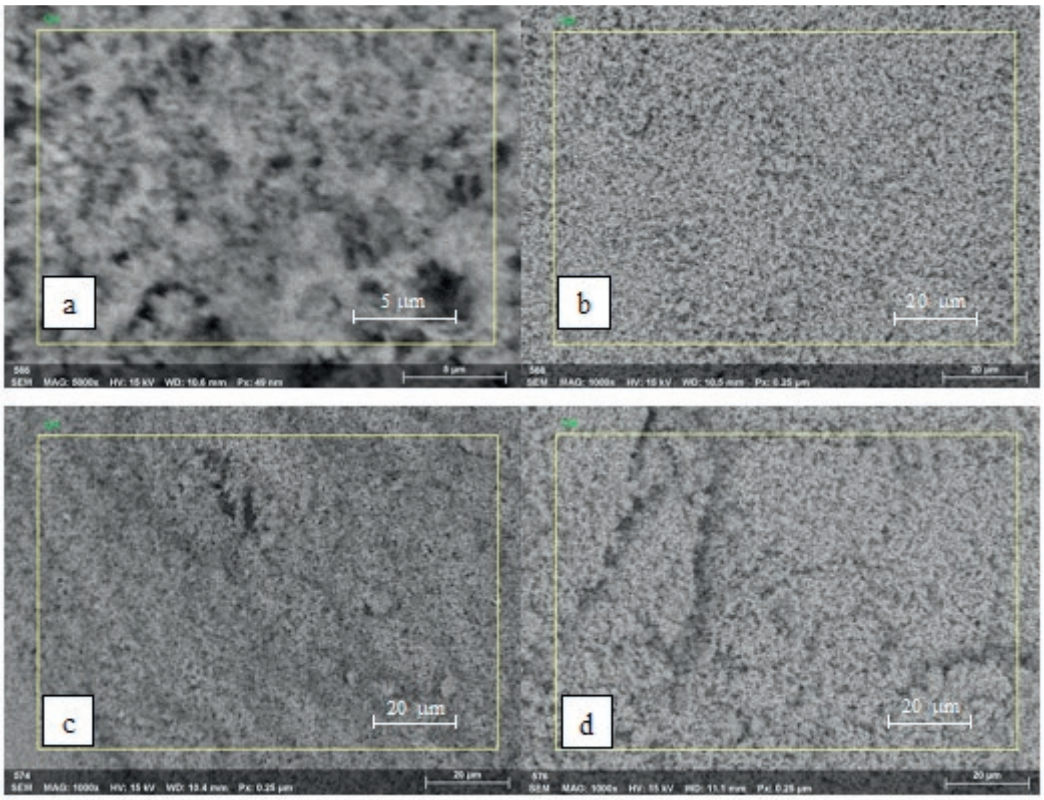

Fig. 1. SEM images of samples: a) – pure ZnO; b) – ZnO-Ag-0.2; c) – ZnO-Ag-0.5; d) – ZnO-Ag-2

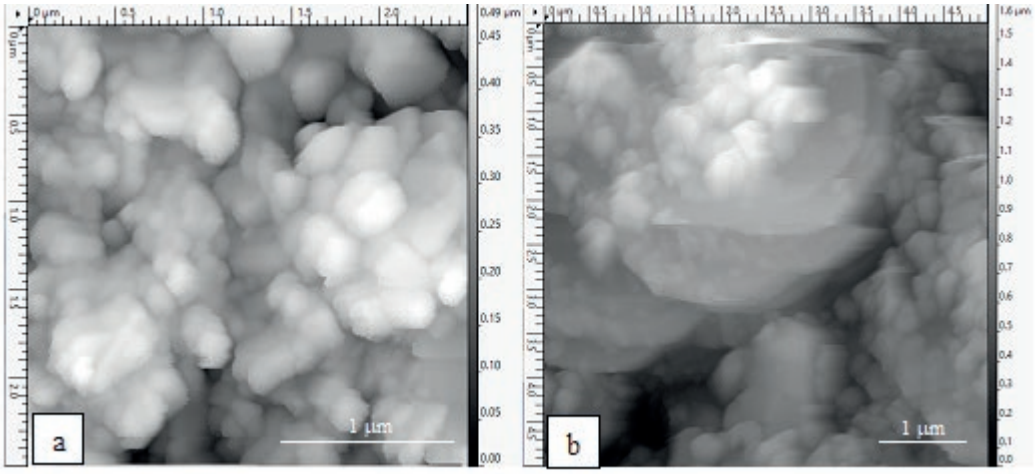

Fig. 2. AFM scan of the sample surface ZnO-Ag-2. Scan area a) – 2.5x2.5 mm, b) – 5x5 mm

Energy dispersive analysis (EDX) was performed to confirm the presence of silver on the surface of the synthesized specimens. Local chemical composition was estimated from EDX spectra of the synthesized specimens shown in Fig. 3. In EDX spectrum for pure ZnO specimen (Fig. 3a) the major elements Zn, O are well distinguished, yet C, Al and Si are also present. Presence of carbon, aluminum and silicon is due to the peculiarities of the substrate on which the specimens were deposited.

The EDX spectrum of ZnO-Ag-0.2 specimen is shown in Fig. 3b. The spectral pattern of the specimen demonstrates that in addition to C and Al impurities, characteristic X-ray lines corresponding to Ag (about 0.25 KeV and 3 KeV) are observed. EDX spectra of the remaining specimens also show the presence of silver

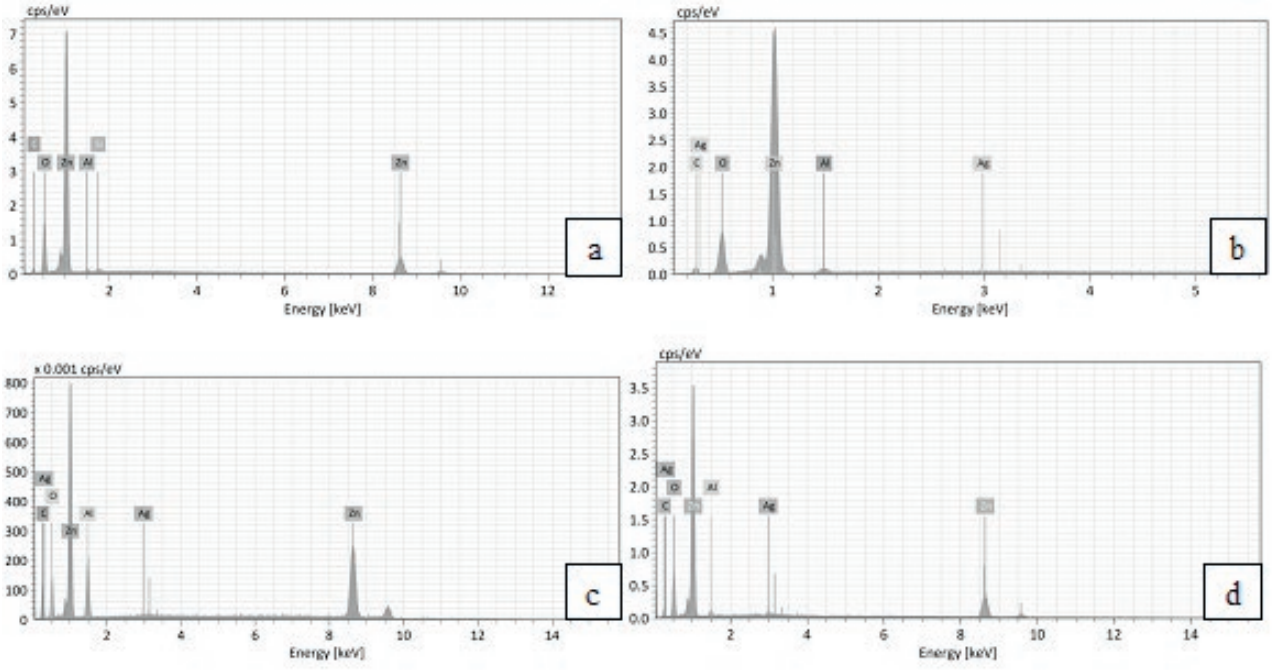

Fig. 3. Energy dispersive spectra EDX a) – pure ZnO; b) – ZnO-Ag-0.2; c) – ZnO-Ag-0.5; d) – ZnO-Ag-2

Table 2. Percentage of composite elements according to EDX spectra

Sample/element	Zn (wt %)	O (wt %)	Ag (wt %)
ZnO	77	23	–
ZnO-Ag-0.2	86	15	0.13
ZnO-Ag-0.5	86.65	13	0.35
ZnO-Ag-2	80.5	17.12	2.38

in the obtained specimens (Fig. 3c, d). The specimens were quantitatively analyzed based on the obtained spectra. The calculated values of mass percentages of elements are given in Table 2.

By means of elemental mapping of ZnO-Ag-0.2 specimen it was possible to evaluate the uniformity of Ag nanoparticles recovery (Fig. 4). The obtained elemental map confirms the presence of homogeneously distributed silver on the substrate surface.

According to the obtained mass percentages of the composite elements (Table 2) calculated on the basis of EDX spectra, it can be noted that the composition of the synthesized specimens corresponds to estimated composition. The obtained data allow to reliably identify the relationship between the change in the photocatalytic activity of the composite and the amount of deposited silver.

3.2. Evaluation of photocatalytic activity of specimens

Activity of the specimens was evaluated on the basis of photocatalytic decomposition of Rhodamine 6G (R6G). The initial concentration of organic dye was 21±0.64 μmol/liter and the volume of solution was 75 mL. The change in dye concentration was evaluated using a PE 5400UV spectrophotometer. Rhodamine 6G has a pronounced absorption peak (526 nm) and its intensity was taken to calculate the concentration using the Bouguer-Lambert-Bera law. The spectral response of Rhodamine 6G was measured 5 and 10 minutes after the beginning of irradiation of the catalyst with UV light. A UV diode with a wavelength of 365 nm and a power of 4.9 W was taken as an irradiation source. The photocatalytic decomposition was investigated under constant stirring. The mass of the catalyst was 5.3±0.2 mg. The study was carried out at least three times for each of the

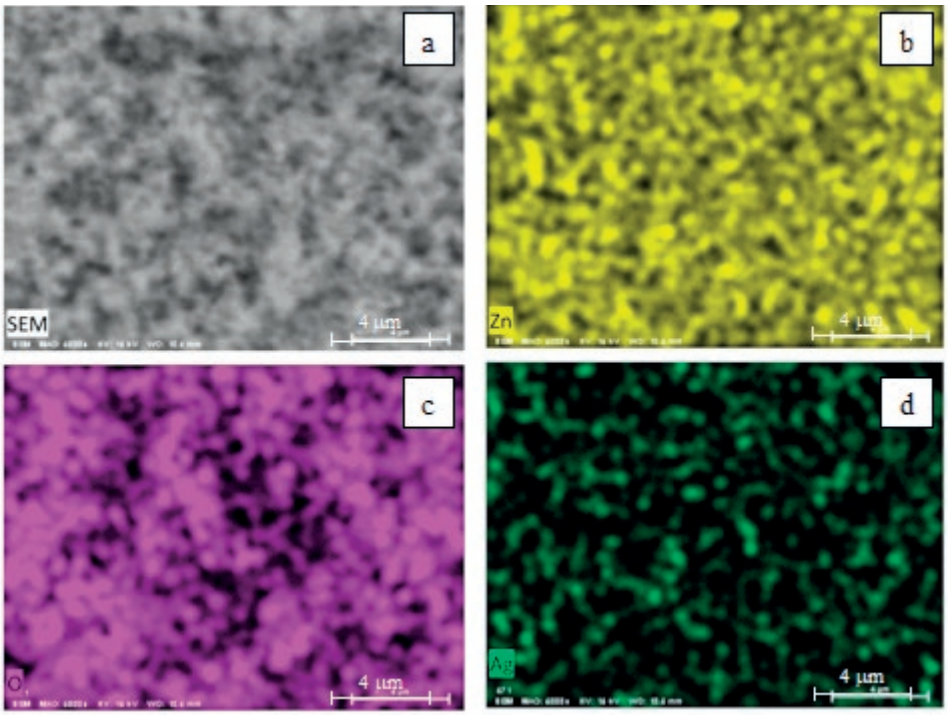

Fig. 4. Elemental map of ZnO-Ag-0.2: a) – SEM image of the surface; map of element concentrations: b) – Zn; c) – O; d) – Ag

specimens to obtain more accurate data on their activity.

Since the same commercial zinc oxide with the same specific surface area was taken as a basis for synthesized catalysts, the weight activity was used as a comparative characteristic. Table 3 shows the results of calculations.

Analysis of the obtained results showed that the average activity of the composite material exceeds the activity of the initial substrate by 58 % (ZnO-Ag-0.2 specimen). As the amount of deposited silver increases from 0.2 to 2 %, the activity of the composite increases from 58 to 92 %. This dependence has a nonlinear character and begins to reach saturation after 2 wt % of precipitated silver (Fig. 5). According to the obtained dependence, it can be noted that further increase in the amount of silver does not lead to a significant increase in the activity of the composite material. On the contrary, the probability of undesirable overlapping of active catalytic centers of zinc oxide increases.

To obtain more complete data on synthesized catalysts, the reaction kinetics was studied. The kinetics of photodegradation of dyes is usually described by the Langmuir-Hinshelwood model and approximated by the following kinetic equation:

$$-dC/dt = k_1 K_a C1 + K_a C,$$

where C is the current dye concentration at time t, k_1 is the rate constant of the process, K_a is the adsorption equilibrium constant.

At low dye concentration ($C \ll 1$ mM), the equation simplifies to a pseudo-first-order process rate equation:

Table 3. Summary table of the results of the study of the activity of samples

Sample	Weight activity (300 сек), mmol/g·s	Weight acrivity (600 сек), mmol/g·s	Rate constant, min^{-1}
ZnO	0.3447	0.2348	0.0660
ZnO-Ag-0.2	0.5461	0.3330	0.1030
ZnO-Ag-0.5	0.6265	0.3648	0.1350
ZnO-Ag-2	0.6611	0.3945	0.1631

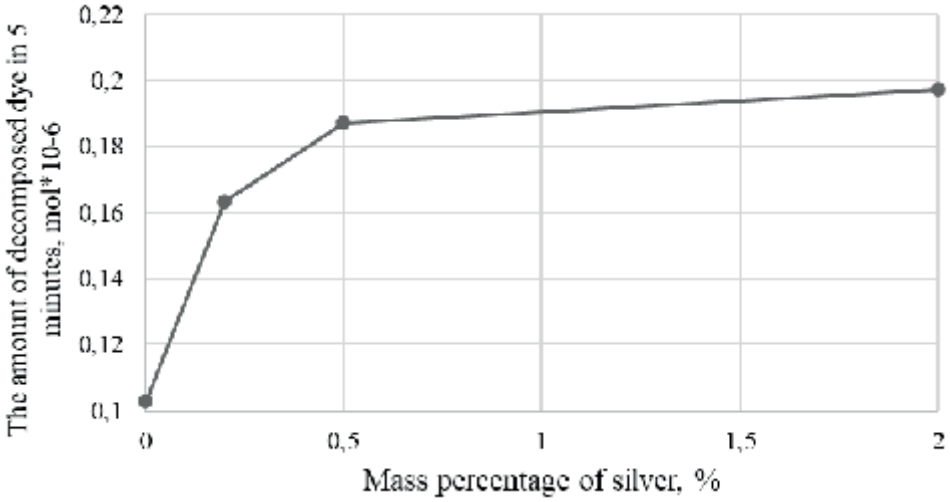

Fig. 5. Graph of the dependence of the composite activity on the silver content

$\ln(C/C_0) = k_1 K_a t = k_{app} t$,

where k_{app} is the pseudo-first-order rate constant.

Fig. 6 shows the graph of dependence $\ln(C/C_0) = f(t)$, plotted on the basis of experimental data. Using linear approximation, the rate constants for the specimens under study were determined, and the estimated values were recorded in Table 3. Since the rate constant is numerically equal to the reaction rate, the value of the constant allows to evaluate the effect of the catalyst on the rate of the dye photodegradation process. Based on the data obtained, it can be said that the deposition of 0.2 wt % silver leads to acceleration of the photocatalytic degradation reaction by 56 %, and when 2 wt % is reached, the rate increases by 147 %.

4. Conclusions

In this course of the present research, specimens of composites with different silver content on their surface were synthesized. The amount of silver in the composite was estimated by EDX spectra. The obtained results are compared with the estimated values and allow

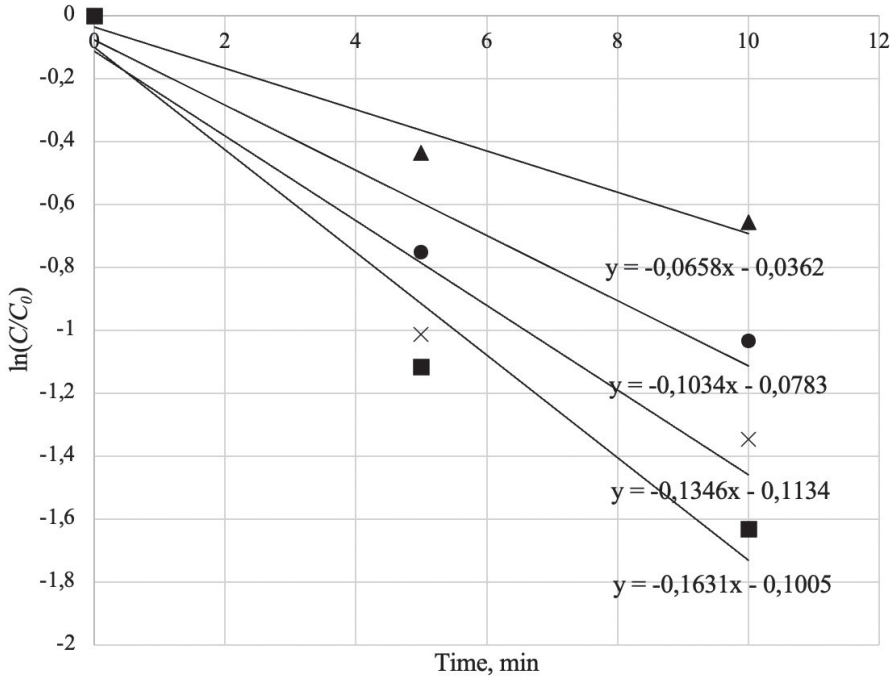

Fig. 6. Dependence graph $\ln(C/C_0) = f(t)$

to reveal the correlation between the amount of silver and the change in the activity of the composite. To confirm the uniformity of silver recovery, an elemental map of the ZnO-Ag-0.2 specimen was obtained.

Analysis of the activity of composites containing silver showed a positive effect of deposited silver on the properties of zinc oxide. An increase in catalytic activity was observed with an increasing amount of silver. The ZnO-Ag-2 specimen has a weight activity higher by 92 % compared to the original ZnO and increases the rate of photocatalytic degradation by 147 %. According to the available dependence of activity on the amount of silver, it can be noted that 2 wt % is the optimum limiting value at which a significant increase in the activity of the composite occurs.

Contribution of the authors

D. G. Radaykin responsible for conducting the experiment, writing the review, final conclusions. V. A. Moshnikov responsible for supervising, setting tasks and discussion of results, text editing.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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