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Photocatalytic activity of zinc oxide – graphene oxide composites

Recently, the use of various dyes in the cellulose, textile, plastics and rubber industries has resulted in severe toxic and dye contamination of wastewater. These toxic dyes are not biodegradable and have a detrimental effect on the environment, blocking sunlight and reducing photosynthesis. Consequently, there is an urgent need to develop a low-cost effective technology for purifying wastewater from harmful organic pollutants. In this context, photocatalysis is an advanced, environmentally friendly oxidation process for wastewater treatment. Recent studies have shown that the use of semiconductor photocatalytically active materials with a wide band gap is an effective method for the decomposition of organic pollutants in aqueous solutions. In this work, a low-cost technology for the synthesis of composite semiconductor zinc oxide / graphene oxide (ZnO / GO) materials by chemical deposition from an aqueous solution has been developed. The morphology, structural and photoluminescent properties of the synthesized ZnO/GO samples with different graphene oxide concentration have been studied. The research of the photocatalytic activity of synthesized zinc oxide – graphene oxide composite materials is carried out by observing an aqueous solution of rhodamine B dye degradation under ultraviolet illumination.

Keywords: chemical deposition, zinc oxide, graphene oxide, composites, optical and structural properties, photocatalytic activity, rhodamine B.

Introduction

In recent years, as a result of environmental changes associated with the ineffective use of water resources, the problem of purifying wastewater from industrial and agricultural waste has become urgent [1, 2]. Numerous waste dyes used in various industries, such as the production of cosmetics, textiles, and food end up in water bodies in the form of wastewater [3–5]. Due to that, emerged the urgent task to develop effective and economical methods for eliminating organic pollution [6, 7]. Various types of biological, physical, and chemical methods, such as chemical precipitation, separation, coagulation, removal, adsorption of activated carbon, chlorination and ozonation, are used for industrial wastewater treatment [8–10]. Chemical methods of chlorination and ozonation are slow processes and as consequence, they are not economically feasible; therefore, the use of these approaches is limited [11]. Traditional filtration methods do not have high efficiency and cause only phase changes in pollutants. In recent decades, photocatalytic decomposition processes have been actively considered successors to the previous methods, since they can significantly accelerate the degradation of organic pollutants [12–14]. Photocatalysis is a reaction in which light is used to activate a substance that changes the rate of a chemical reaction. Under the illumination of semiconductor, photocatalyst electrons move to the conduction band, a hole appears in the valence band, thus an electron-hole pair is generated. The generated electron-hole pairs cause a complex series of secondary reactions with dye molecules, which leads to the complete degradation of dye contaminants adsorbed on the semiconductor surface [15].

Zinc oxide (ZnO) is one of the most studied photocatalytically active materials due to its low toxicity, wide band gap (3.37 eV), long-term stability, high photostability, and high efficiency [16, 17]. Usually, oxide semiconductors have relatively low charge mobility and a high degree of electron-hole recombination, which reduces the efficiency of the photocatalytic reaction and prevents their widespread use as practical photocatalysts [18].

The photocatalytic characteristics of ZnO can be improved by synthesizing composites based on it in combination with other components, such as noble metals [19], carbon materials [20, 21], and by doping [22]. In particular, nanocomposites with graphene and graphene oxide (GO) have attracted the attention of researchers in recent years. ZnO is a good electron donor with high optical activity and stability. Moreover, graphene is a good electron acceptor with high conductivity. Thus, the hybrid system can act conjointly by

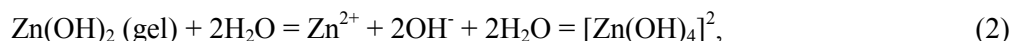
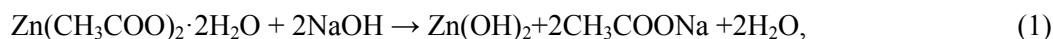
increasing the migration efficiency of photo-generated electrons and decreasing recombination. H.Fan et al. synthesized a series of composites of graphene and ZnO by a simple hydrothermal method [23]. The effect of the graphene inclusion in ZnO structure on the photocatalytic degradation of an aqueous dye solution was noted in the article [24]. In most of these works, ZnO / graphene oxide composites were synthesized either at a higher temperature or with complex equipment and a complex reaction procedure with the participation of intermediate products of the growth of zinc oxide particles on reduced graphene oxide sheets is required.

In this regard, the task of improving photocatalytic characteristics by the efficient, low-cost synthesis methods of composite materials based on ZnO is urgent.

Experimental

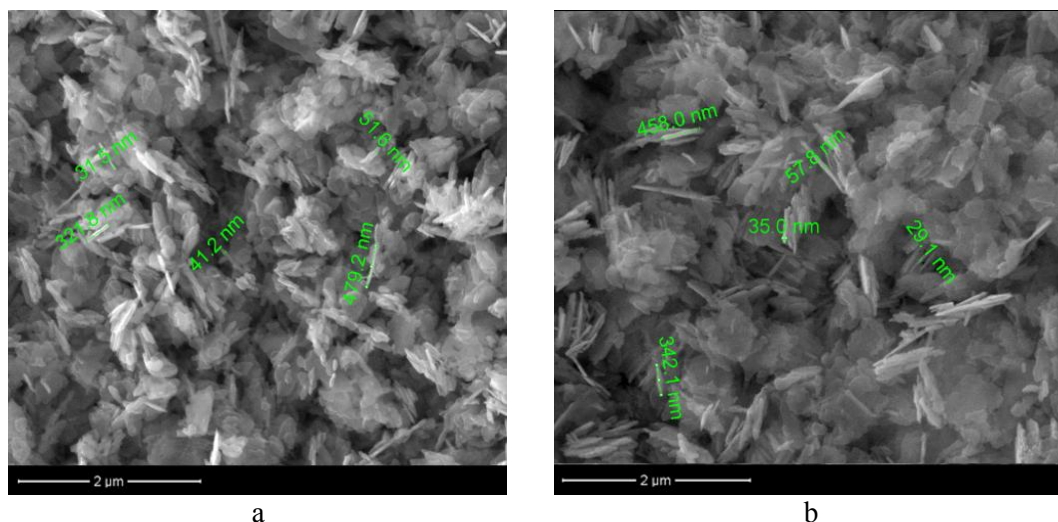
Composite materials based on zinc oxide and graphene oxide were synthesized by low-temperature chemical deposition from an aqueous solution. For the synthesis of ZnO / GO composites, we used ready graphene oxide obtained by the Hammers method [25]. Graphene oxide was dissolved in distilled water for half an hour using an ultrasonic bath. In a separate glass, zinc acetate dihydrate ($(\text{CH}_3\text{COO})_2\text{Zn}\cdot 2\text{H}_2\text{O}$, Sigma-Aldrich) was dissolved in distilled water. Then both solutions were mixed on a magnetic stirrer. Then, to form zinc oxide nanoparticles, an aqueous solution of sodium hydroxide (NaOH, Sigma-Aldrich) was added dropwise to a glass beaker with an aqueous solution of zinc acetate and graphene oxide. Then the entire solution was thoroughly mixed on a magnetic stirrer for another 15 minutes. The whole process of synthesis of ZnO composites - GO was carried out at room temperature.

The NaOH concentration was 0.7M with a zinc acetate concentration of 50mM. The graphene oxide concentration was varied from 0.01 to 0.7 mm wt% (samples: ZGP 2 – 0.01 wt% GO, ZGP 4 – 0.025 wt%, ZGP 5 – 0,14 wt% GO, ZGP 7 – 0,7 wt%). The resulting precipitate was washed with distilled water, separated by centrifugation, and then dried in an oven at 125 °C for 12 hours. The formation of zinc oxide in this process can be described by the following chemical reactions [26]:



Results and Discussion

The results of electron microscopy studies carried out on a Quanta 200i 3D scanning electron microscope (FEI Company) with a directly heated tungsten cathode showed that the ZnO / GO composites synthesized by this growth technique are two-dimensional structures in the form of 2D thin petals about 30 nm thick (Figure 1). It was noted that an increase in the concentration of GO in the growth solution of ZnO / GO leads to the decrease in the thickness of ZnO petals. As a result, the 2D structures become thinner, the amount of the flocculent structure increases. It allows increasing their working specific surface to equal weight.



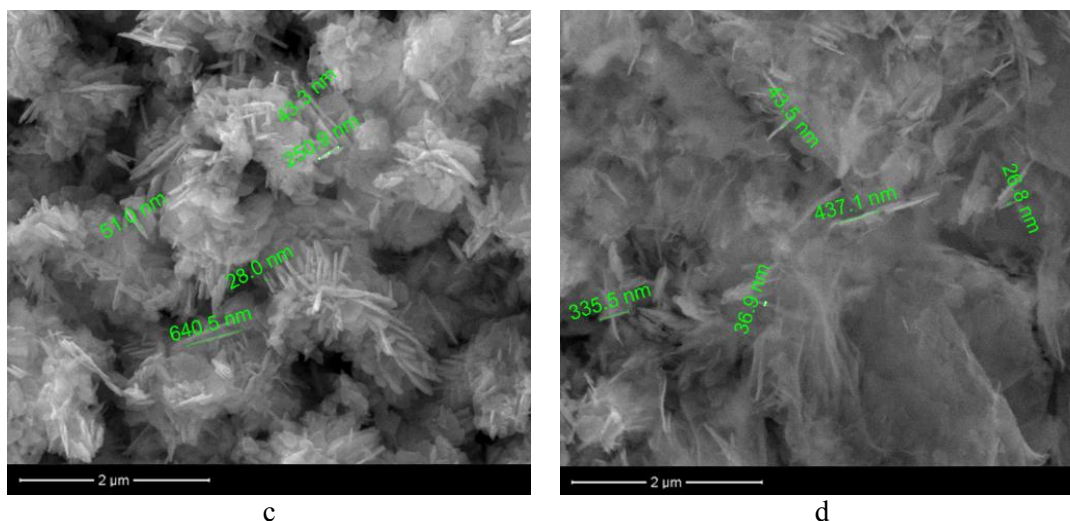


Figure 1. Morphology of ZnO / GO samples: a – ZGP 2 – 0.01 wt% GO, b – ZGP 4 – 0.025 wt% GO, c – ZGP 5 – 0,14 wt% GO, d – ZGP 7 – 0.7 wt% GO.

The structural properties of the synthesized ZnO / GO samples were studied by X-ray diffraction analysis. X-ray diffractometry measurements were performed under the same conditions for all synthesized samples. Figures 2, 3 show diffractograms of the samples ZGP 2 and ZGP 7 with the lowest and highest concentration of graphene oxide, respectively, in considered series of ZnO / GO samples.

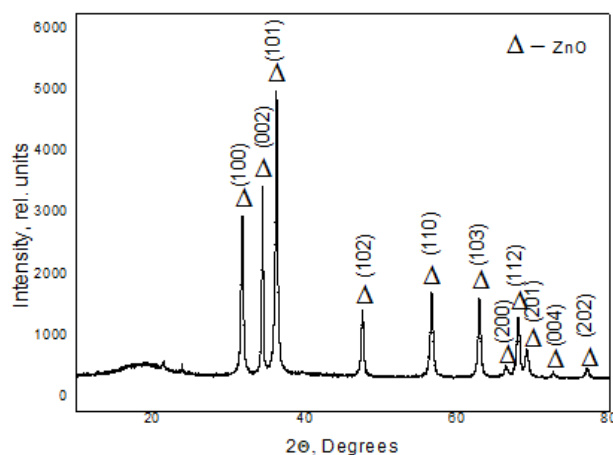


Figure 2. X-ray diffraction of ZGP 2 sample

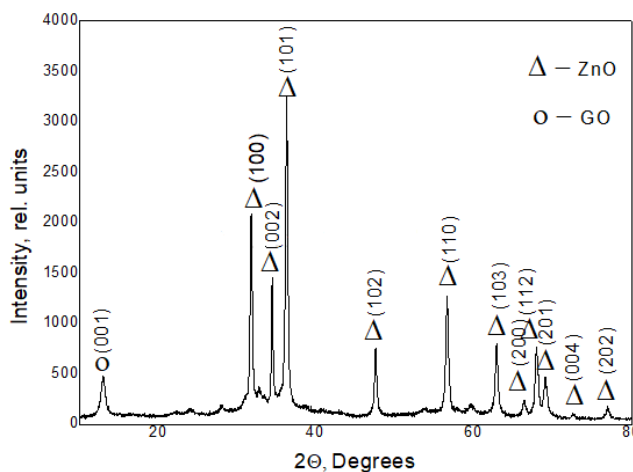


Figure 3. X-ray diffraction of ZGP 7 sample

All ZnO / GO samples exhibit the hexagonal structure of zinc oxide wurtzite (JCPDS card # 01-080-3004). The diffractogram of the ZGP 7 sample with the highest GO concentration contains (001) reflex, corresponding to GO sheets.

Changes in the structural properties of ZnO / GO composites depending on the concentration of graphene oxide in the growth solution were also studied using Raman spectroscopy (Figure 4).

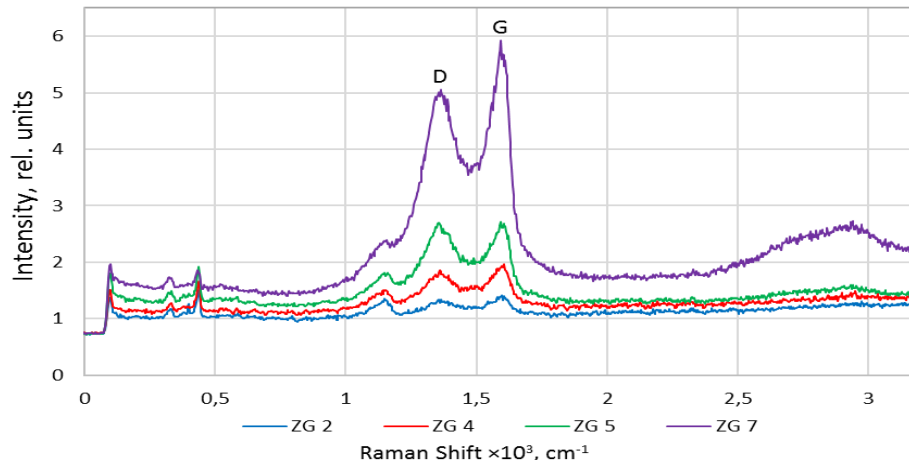


Figure 4. Raman spectra of ZnO / GO samples: ZGP 2 – 0.01 wt% GO, ZGP 4 – 0.025 wt% GO, ZGP 5 – 0,14 wt% GO, ZGP 7 – 0.7 wt% GO.

Raman spectra allow one to take into account conjugated and double carbon-carbon bonds, which leads to high-intensity peaks in the Raman spectrum. A typical GO Raman spectrum is characterized by a G band at a displacement of the 1605 cm^{-1} , which corresponds to the E_{2g} phonon of sp^2 carbon atoms, and a D band at 1353 cm^{-1} , which corresponds to the breathing mode of point phonons with A_{1g} symmetry.

The Raman spectra of synthesized ZnO / GO composites show that the D peaks for these samples appear at 1370 cm^{-1} . G peaks at 1600 cm^{-1} characterize lattice distortions. A shift in the D band indicates a change that can occur due to certain defects, such as vacancies, grain boundaries [27] and amorphous forms of carbon [28]. The intensity ratio I_D/I_G was $0.9 / 1$. The study [29] demonstrated that an increase in the I_D/I_G ratio of graphene oxide indicates an increase in the number of defects in the structure. The lowest ratio $I_D/I_G = 0.85$ was observed for ZGP 7 sample, which indicates the quality of this sample.

Graphene, a two-dimensional planar monolayer of carbon, whose atoms are connected in a honeycomb lattice structure, possesses such electronic properties as a zero band gap and high conductivity, which in combination with photocatalytically active ZnO should help to reduce the recombination of photogenerated electron-hole pairs and lead to an increase in the efficiency of photoconversion. Thus, graphene oxide in ZnO / GO composites acts as a fast electron transfer channel. In addition, the presence of oxygen-containing functional groups of graphene oxide makes it an excellent support material for anchoring metal oxide particles during synthesis. Figure 5 illustrates a diagram of a possible illustration of the degradation process of the RhB dye under the influence of ultraviolet radiation in the presence of ZnO / GO samples.

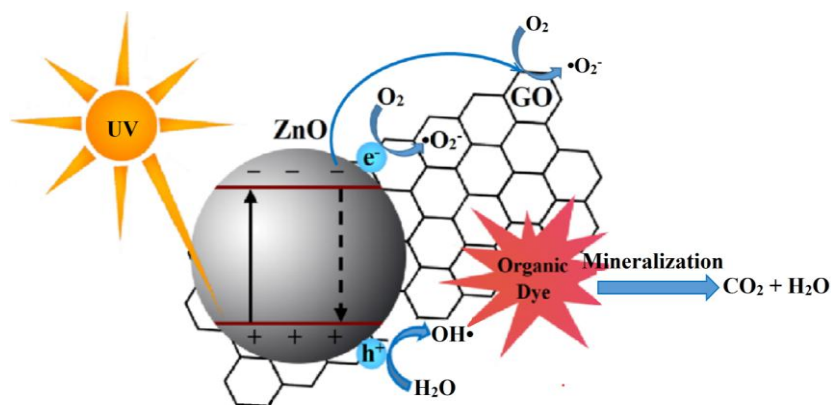


Figure 5. Possible charge transfer circuit in the ZnO / GO samples

Under the influence of UV radiation, electrons e^- move from the upper levels of the valence band (VB) of semiconductor materials to the conduction band (CB). These photogenerated electrons are absorbed by oxygen O_2 , resulting in the superoxide anion O_2^- . At the same time, as a result of protonation, HOO^\bullet radicals are obtained. Holes h^+ , located in the valence band, move to the surface of ZnO and GO, and interact with either water (H_2O) or OH^- , which leads to the production of such active hydroxyl substances as OH^\bullet , which, together with HOO^\bullet radicals, decompose the dye into components. Thus, the process of photogeneration of e^- - h^+ electron-hole pairs on the surface of ZnO / GO composites with a large specific surface area leads to degradation of the rhodamine B (RhB) dye.

To study the photocatalytic activity of the synthesized ZnO / GO composites, we used an aqueous solution of rhodamine B, containing 8 ml of the dye dissolved in 500 ml of distilled water on a magnetic stirrer for 30 minutes. Then, 9 mg of the obtained ZnO / GO composites were added to 112.5 ml of the solution, followed by thorough mixing. The measurement of photocatalytic degradation was carried out in a glass reactor. The source of UV illumination was a mercury lamp (LIH Germany, power 14 W), which was located inside the reactor with the solution.

The photocatalytic activity of the synthesized ZnO / GO composites was evaluated by observing the change in the optical density spectra of the RhB dye solution in the presence of ZnO / GO samples exposed to UV radiation for 150 minutes. The solution was sampled every 30 minutes (Figure 6).

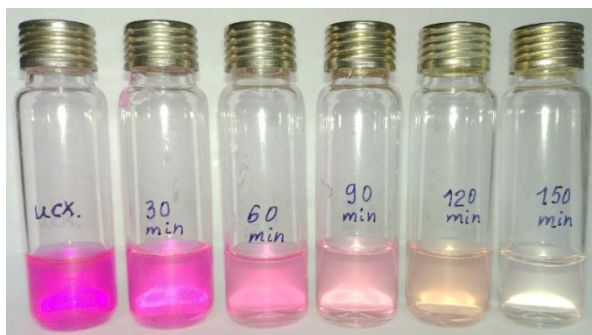
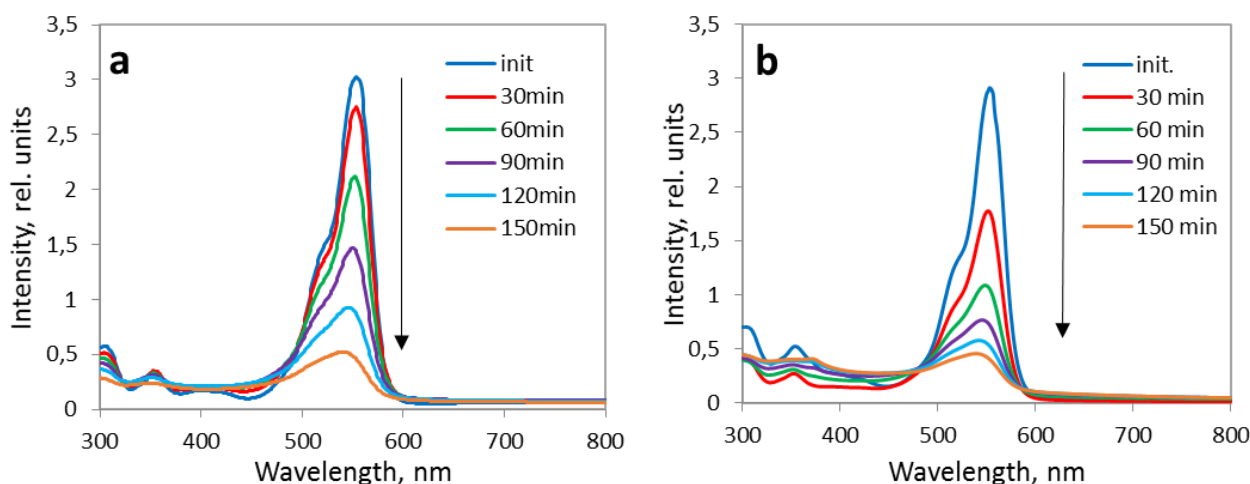


Figure 6. Photo of the RhB solution in the presence of the ZGP 7 sample subjected to UV illumination

Figure 7 represents optical density spectra during the degradation of an aqueous solution of a dye in the presence of ZnO / GO composites under ultraviolet illumination. The results showed that the maximum value of the absorption intensity for the initial dye solution with ZnO / GO composites is at 555 nm. It is noted that, with an increase in the UV exposure time, the absorption intensity of rhodamine B in the presence of ZnO / GO samples decreases, which indicates a decrease in the concentration of RhB in the solution.



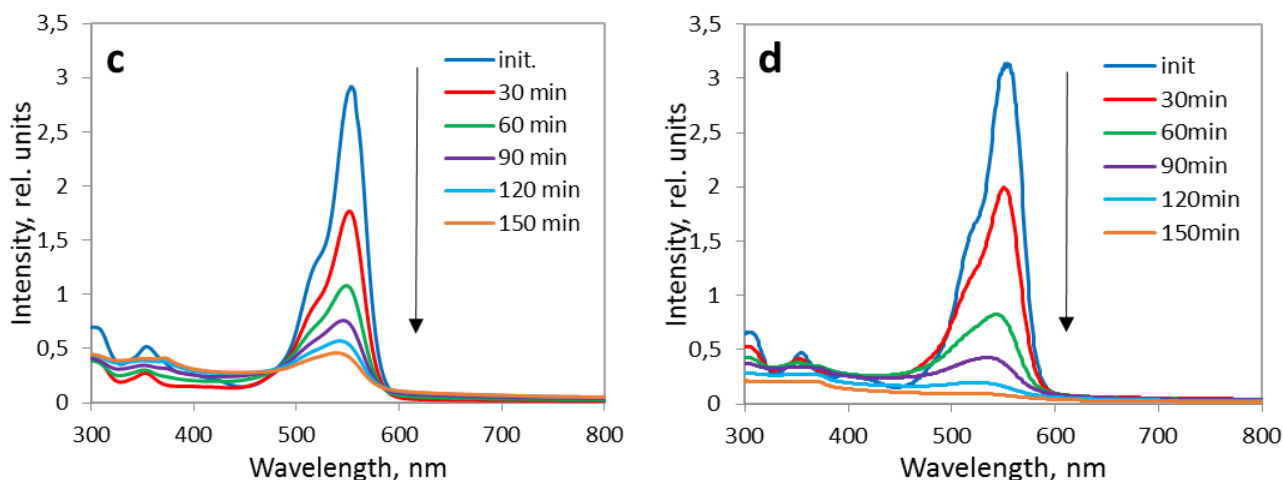


Figure 7. Optical density spectra of an aqueous solution of RhB in the presence of samples: a – ZGP 2, b – ZGP 4, c – ZGP 5, d – ZGP 7.

To compare the photocatalytic activity of ZnO / GO powders with different GO concentration in the growth solution under degradation of an aqueous dye solution, kinetic curves were presented in Figure 8.

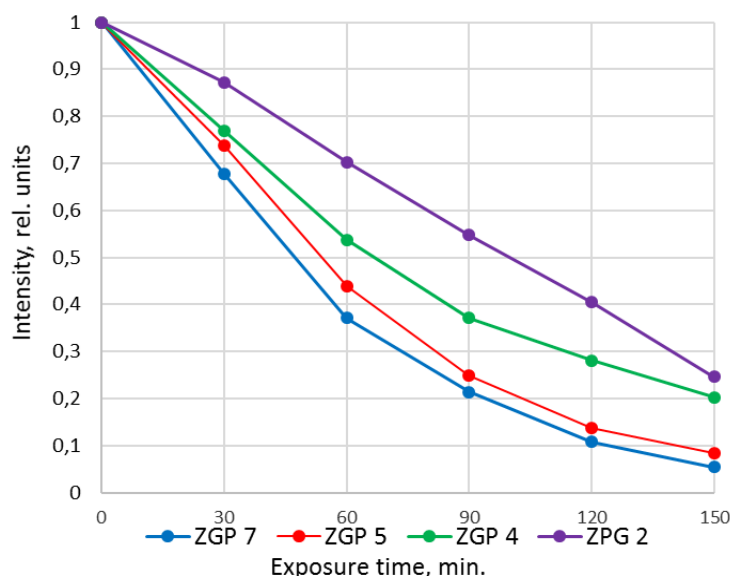


Figure 8. Kinetic curves of RhB degradation in the presence of ZnO / GO composites with different concentrations of GO: ZGP 2 – 0.01 wt% GO, ZGP 4 – 0.025 wt% GO, ZGP 5 – 0.14 wt% GO, ZGP 7 – 0.7 wt% GO.

It was noted that an increase in the concentration of GO in the growth solution makes it possible to obtain more photocatalytically active composites ZnO-GO because an increase in the GO concentration in the growth solution provides an increase in the specific surface area of the composites. Moreover, this increase promotes a decrease in the recombination of photogenerated electrons, which leads to an increase in active $\text{HOO}\cdot$ radicals in solution under UV radiation and accelerates the decomposition of the organic dye.

For ZnO / GO samples with different GO concentrations, photoluminescence spectra were investigated (Figure 9). Data on the photoluminescent properties of photocatalytically active zinc oxide were published in a paper [30]. The PL spectra of all the samples consisted of a narrow (~ 0.25 eV) NBE at ~ 380 nm, and broad deep-level emission (DLE) in the range from 450 to 750 nm with a maximum of around 555 nm. It is suggested that the origin of the violet emission at $\lambda \sim 420$ nm (2.96 eV) is due to the transitions from the conduction band (CB) to the holes localized at the defect level associated with zinc vacancy (VZn) [31].

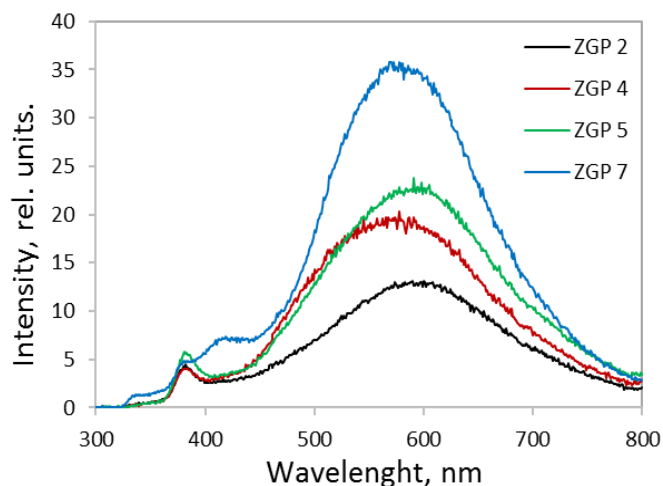


Figure 9. Photoluminescence of ZnO / GO samples with different GO concentration: ZGP 2 – 0.01 wt% GO, ZGP 4 – 0.025 wt% GO, ZGP 5 – 0.14 wt% GO, ZGP 7 – 0.7 wt% GO.

As can be seen from Figure 9, the highest DLE photoluminescence is observed for the ZGP 7 sample, which contains the largest amount of graphene oxide in the growth solution (0.7 wt % GO). It is noted that a decrease in the GO concentration leads to a decrease in the DLE photoluminescence.

Conclusions

Low-cost synthesis of ZnO / GO nanocomposites by chemical deposition from the solution was developed. The photocatalytic activity, morphology, structural and photoluminescent properties of the synthesized samples were studied. The results of electron microscopy studies have shown that the obtained composites consist of 2D petals about 30 nm thick. At the same time, an increase in the concentration of GO in the growth solution of ZnO / GO does not significantly change their morphology. A study of the structural properties of the synthesized ZnO / GO composites showed that the Raman spectra contain D peaks at displacement of the 1370 cm^{-1} , and G peaks at 1600 cm^{-1} . The intensity ratio ID / IG was 0.9 / 1. The smallest ratio ID / IG = 0.85 for the ZGP 7 sample, which contains the largest amount of graphene oxide in the growth solution (0.7 wt% GO), indicates the quality of this sample. It is noted that the highest DLE photoluminescence is observed for the ZGP 7 sample. In addition, an increase in the GO concentration leads to an increase in DLE photoluminescence. When studying the photocatalytic activity of the obtained samples regarding the decomposition of the dye rhodamine B (RhB), it was noted that an increase in the concentration of GO in the growth solution makes it possible to obtain more photocatalytically active composites ZnO-GO.

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Мырыш оксиді–графен оксиді композиттердің фотокаталитикалық белсенділігі

Соңғы кездері целлюлоза, тоқыма, пластмасса және резеңке өнеркәсібінде әр түрлі бояғыштарды қолдану ағынды сулардың улы заттармен және бояғыштармен қатты ластануына әкелді. Бұл улы бояғыштар күн сәулесін бөгеп, фотосинтезді азайтып, қоршаған ортаға зиянды әсер етеді. Сондықтан ағынды суларды зиянды органикалық ластаушы заттардан тазартудың аз шығынды тиімді технологиясын тез арада әзірлеу қажет. Осы тұрғыда фотокатализ ағынды суларды тазартуға арналған жетілдірілген, экологиялық таза тотығу процесі болып табылады. Соңғы зерттеулер көрсеткендей, кен шектеу аймағы бар жартылай өткізгішті фотокаталитикалық белсенді материалдарды қолдану сулы ерітінділердегі органикалық ластаушы заттардың ыдырауының тиімді әдісі болып табылады. Осы мақалада композициялық жартылай өткізгіш материалдарды сулы ерітіндіден химиялық тұндыру арқылы мырыш оксиді/графен оксидін (ZnO/GO) синтездеудің арзан әдісі әзірленген. Графен оксидінің әр түрлі концентрациясы бар синтезделген ZnO/GO үлгілерінің морфологиясы, құрылымдық және фотолуминесценттік қасиеттері зерттелді. Синтезделген композициялық материалдардың фотокаталитикалық белсенділігін зерттеу ультракүлгін сәулелену кезінде родамин Б бояғышының сулы ерітіндісінің ыдырауын бақылау арқылы жүзеге асырылды.

Кілт сөздер: химиялық тұндыру, мырыш оксиді, графен оксиді, композиттер, оптикалық және құрылымдық қасиеттері, фотокаталитикалық белсенділік, родамин Б.

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Фотокаталитическая активность композитов оксида цинка–оксида графена

В последнее время использование различных красителей в целлюлозной, текстильной, пластмассовой и резиновой промышленности привело к сильному загрязнению сточных вод токсичными веществами и красителями. Эти токсичные красители не поддаются биологическому разложению и оказывают пагубное воздействие на окружающую среду, блокируя солнечный свет и снижая фотосинтез. Следовательно, существует острая необходимость в разработке малозатратной эффективной технологии очистки сточных вод от вредных органических загрязнителей. В этом контексте фотокатализ является перспективным, экологически безопасным процессом окисления для очистки сточных вод. Недавние исследования показали, что применение полупроводниковых фотокаталитически активных материалов с широкой запрещенной зоной является эффективным методом разложения органических загрязнителей в водных растворах. В настоящей статье разработана малозатратная технология синтеза композиционных полупроводниковых материалов оксид цинка/оксид графена (ZnO/GO) методом химического осаждения из водного раствора. Исследованы морфология, структурные и фотолуминесцентные свойства синтезированных образцов ZnO/GO с различной концентрацией оксида графена. Исследование фотокаталитической активности синтезированных композиционных материалов оксид цинка–оксид графена проводили путем наблюдения за деструкцией водного раствора красителя родамин Б при ультрафиолетовом освещении.

Ключевые слова: химическое осаждение, оксид цинка, оксид графена, композиты, оптические и структурные свойства, фотокаталитическая активность, родамин Б.