PHOTOCATALYTIC ACTIVITY OF ZnO/TiO_2 COMPOSITES IN CIRCULATING CONDITIONS

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The development of new efficient photocatalysts is an important task for solving problems related to the purification of water and air from organic pollution. Composite materials based on ZnO and TiO_2 exhibit high photocatalytic activity, which makes them promising for this application. In this work, we present the synthesis and study of the photocatalytic activity of ZnO/TiO_2 composites obtained by the method of intermediate hydroxide deposition. The synthesis was carried out on the surface of Evonik P25 TiO_2 with three different mass ratios of ZnO to TiO_2: 1:3, 1:1, and 3:1. The resulting composites were studied by X-ray diffraction (XRD), spectroscopy to determine the optical band gap, and subjected to photocatalytic decomposition under circulating conditions. It was confirmed by XRD that ZnO crystallizes in the wurtzite phase of hexagonal syngony, and TiO_2 is contained in the form of two modifications: anatase and rutile. The effect of the mass ratio of ZnO to TiO_2 on the optical band gap has been studied. The optical band gap of ZnO/TiO_2 composites was determined using the Kubelka-Munk algorithm. For the composites (1)ZnO/TiO_2 and (3)ZnO/TiO_2, the bandgap was 3.22 eV, and the lowest value (2.99 eV) was obtained for the composite with an equal ratio of ZnO to TiO_2 - (2)ZnO/TiO2. The photocatalytic activity of ZnO/TiO2 composites was studied under circulating conditions with congo red dye in the presence of four different composite weights: 0.2, 0.4, 0.6, and 2 g. The maximum efficiency of photocatalytic decomposition of the dye was observed for the composite with an equal ratio of ZnO to TiO_2 at a dosage of 2 g of the composite per 0.075 g of dye. The synthesized ZnO/TiO_2 composites exhibit high photocatalytic activity, which makes them promising materials for water and air purification from organic pollution. The optimum mass ratio of ZnO to TiO_2 for the photocatalytic decomposition of congo red dye is 1:1.

Keywords: *composites, congo red, dye, photocatalyst, titanium dioxide, zinc oxide.*

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1. Introduction

Water pollution has become one of the most pressing issues of our time due to population growth (Qi et al., 2020; Samadi et al., 2016). As population growth coincides with an increase in the production of synthetic compounds, in the textile and paint or food industries, the increase in emissions of these compounds without any treatment will lead to the generation of large amounts of waste in the environment, threatening human existence and life (Phuruangrat et al., 2019). Industrial enterprises discharge approximately 300-400 million tons of waste into the world's water bodies annually, such as organic dyes, phenolic organic substances, antibiotics, or heavy inorganic metals (Zhan et al., 2022). Hazardous waste management is crucial as it has direct harmful effects on humans, animals and aquatic ecology (Liu et al., 2020; Singh & Soni, 2020).

One of the main sources of water pollution is improper waste disposal, such as the discharge of industrial dyes into water sources. This makes it necessary to constantly search for low-cost, efficient methods of dye removal, as well as other wastewater treatment methods developed in combination with new materials. These methods include photodegradation of dyes using appropriate photocatalysts (Firdaus et al., 2012; Georgakopoulos et al., 2015; Siuleiman et al., 2013). Photocatalytic semiconductor materials have attracted considerable attention as advanced physicochemical methods for the decomposition of organic pollutants (Ahmed et al., 2019). This is due to their low energy consumption, simplicity, and mild reaction course. In removing organic contaminants and disinfecting water from bacteria, semiconducting metal oxides such as TiO_2, ZnO, ZnS, CdS or Fe_2O_3 exhibit excellent photocatalytic and antibacterial activities, making them the most promising materials in this field (Abebe et al., 2020). Among the various semiconducting metal oxides, TiO_2 and ZnO, which have similar band gaps (ZnO, 3.37 eV, and TiO_2, 3.2 eV), are found to be excellent photocatalysts for water purification (Raza et al., 2016). Nevertheless, ZnO is recognized as a promising alternative to TiO_2 due to its high exciton binding energy (60 meV), high electron mobility, high quantum efficiency, oxidation resistance, non-toxicity, biocompatibility, and low cost (Swati et al., 2020).

Composite materials obtained by combining two semiconductors based on their strong respective properties offer excellent combinations for various applications (El Mragui et al., 2019; Tian et al., 2009). ZnO and TiO_2 semiconductors, independently of each other, are of great interest due to their wide range of properties, but these two semiconductors differ in several aspects: ZnO is a direct bandgap semiconductor, while TiO_2 has an indirect junction, has a relatively high absorption rate, and generally has higher photocatalytic activity than ZnO (Lavand & Malghe, 2015; Zhao et al., 2012).

In the case of photocatalytic processes, several important properties of materials must be taken into account, such as their ability to absorb photons, separate and transfer charges, and the rate of charge recombination. This means that not every semiconductor can be effectively used for photocatalysis, as the main problem for most of them is charge recombination (Banerjee et al., 2006). Based on the common characteristics of both materials, such as environmental friendliness, high electronic properties and almost identical band gap energy barriers, a highly efficient binary ZnO/TiO_2 photocatalytic composite can be obtained. Compared to pure ZnO or TiO_2, their combined structures exhibit enhanced photocatalytic activity due to a wider light absorption range, efficient charge separation, reduced electron-hole recombination, and increased charge carrier lifetime, leading to improved charge transfer (Dhanalakshmi et al., 2013).

For the synthesis of ZnO/TiO_2 nanocomposite, various methods such as hydrolysis, chemical vapour deposition, radio frequency magnetron sputtering, spray pyrolysis and sol-gel methods have been used (Hasnidawani et al., 2016; Moradi et al., 2016). In this work, the deposition method was used to prepare the ZnO/TiO_2 composite.

The aim of the work was to study the photocatalytic activity of ZnO/TiO_2 composites in circulating conditions.

The tasks of this work included investigation of the influence of the mass ratio of ZnO to TiO_2 on the crystalline structure, optical properties, and photocatalytic activity of the composites; determination of the optimal dosage of the ZnO/TiO_2 composite photocatalyst for the degradation of Congo Red dye in circulation conditions.

2. Materials and Methods

The synthesis of a ZnO/TiO_2 composite photocatalyst was carried out by the deposition of intermediate hydroxides and their hydrolysis on the surface of Evonik P25 TiO_2 (the amount of powder was different to obtain three different mass ratios of ZnO to TiO_2 ((1)ZnO/TiO_2 - 1:3, (2)ZnO/TiO_2 - 1:1, (3)ZnO/TiO_2 - 3:1)). The synthesis scheme is shown in Fig. 1.

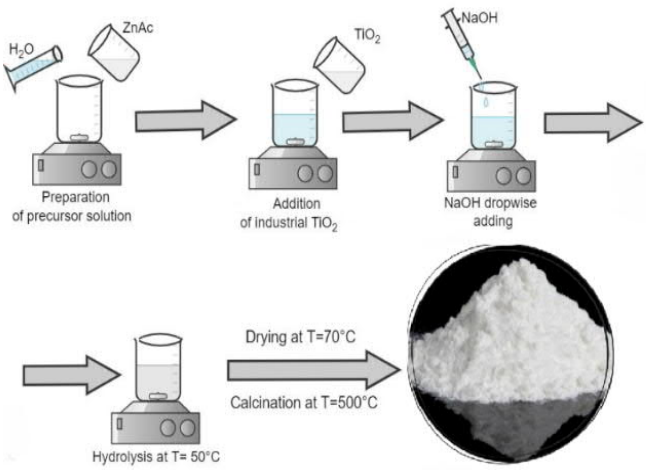

***Fig. 1**. Sequence of stages in the laboratory process of ZnO/TiO_2 composites synthesis*

To obtain the intermediate product $Zn(OH)_2$, a solution precursor of ZnAc (Tech Grade, China) was used, which was precipitated by a 1M solution of NaOH (CAS, Germany).

The obtained photocatalyst was investigated using X-ray diffraction method with a Rigaku TTR3 powder X-ray diffractometer (Japan, with Cu Kα radiation (λ = 1.5406 Å), 30 mA and 30 kV). Additionally, its optical bandgap was analyzed using an inSpect-101UV spectrophotometer (190-1000 nm, Spectrometer65TM, China). The photocatalytic properties were investigated in a 5 L laboratory circulating setup using a UV lamp with a wavelength of 245 nm and a power of 24 W. Filtration was performed using syringe filters with a diameter of 25 mm and a pore size of 0.22 μm (IDEALAB, China).

3. Results and Discussion

The crystalline structure of the synthesized composites was determined using X-ray diffraction method, which allowed for the identification of the most intense peaks of zinc (II) oxide and titanium (IV) oxide. From the data shown in Fig. 2, it can be concluded that zinc (II) oxide was crystallised in the wurtzite phase of hexagonal syngony. Titanium (IV) oxide is contained in the form of two modifications - anatase and rutile in a ratio of approximately 86:14.

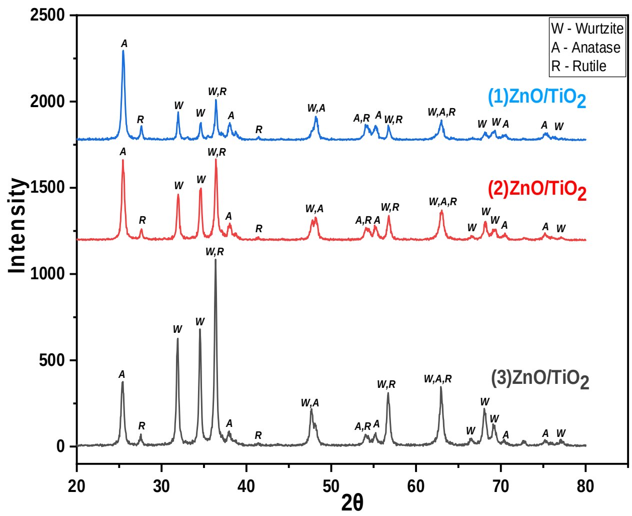

***Fig. 2.** XRD of the obtained ZnO/TiO_2 composites*

As can be seen from XRD, in composite (1), the intensity of the characteristic maximum of anatase is the highest, and the intensity of the maximums of rutile and wurtzite is low. With an increase in the content of the precipitated zinc (II) oxide phase, its characteristic maxima become more intense.

In order to compare the value of the optical bandgap, the absorption spectra of the ZnO/TiO_2 composite photocatalysts were taken. The photocatalyst samples were prepared in distilled water. For this 1 mg of the photocatalyst powder was mixed with 4 mL of distilled water and sonicated for 1 minute to obtain a homogeneous colloidal suspension. After that, the absorption spectrum of the resulting suspension was taken.

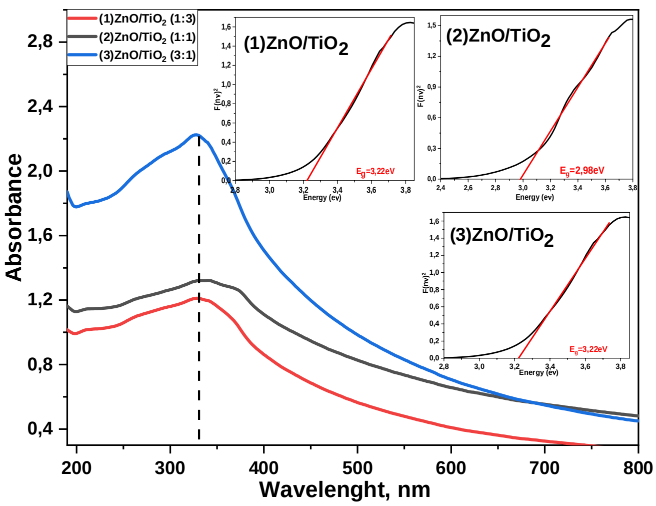

Fig. 3. *Absorbance spectra and Kubelka-Munk diagrams for: (1)ZnO/TiO_2; (2)ZnO/TiO_2; (3)ZnO/TiO_2, annealed at 500 °C*

The results of the conducted research are presented in Fig. 3. From the absorption spectra, was observed that the λ_{max} of all three composites is approximately in the same range, around ~335 nm. Additionally, an increase in absorption intensity was observed, which was associated with the increase in ZnO content in the composites from the lowest ratio (1)ZnO/TiO_2 (1:3) to the highest (3)ZnO/TiO_2 (3:1).

The optical bandgap width was determined using the Kubelka-Munk algorithm as employed in the study (Ivanenko et al., 2021). As seen from the obtained results, the optical bandgap width (Eg) for samples (1)ZnO/TiO_2 and (3)ZnO/TiO_2 is 3.22 eV, while for sample (2)ZnO/TiO_2 it is 2.98 eV. The reduction in the bandgap width of the composite (2)ZnO/TiO_2 with an equal component ratio of 50/50 is attributed to the formation of a bandgap offset at the interface of the two semiconductors and the reaction of average intensity levels due to defects in the crystal lattice, which is also associated with an indirect heterojunction.

Considering the aforementioned, sample (2)ZnO/TiO_2 possesses an optimal bandgap width (2.98 eV) for absorbing visible light and minimizing photo-generated electron-hole recombination, which explains its high degree of photocatalytic degradation.

To assess the photocatalytic properties and determine the maximum effective dosage of the obtained ZnO/TiO_2 composites, an experiment was conducted. In this experiment, various amounts of ZnO/TiO_2 composites were added to a 3 L model solution of Congo Red dye with an initial concentration of 25 mg/L, specifically: 0.2, 0.4, 0.6, and 2.0 g. Additionally, the complete degradation of the dye into simple compounds CO_2 and H_2O was investigated for each sample. The investigation was performed using acid-base titration with evacuated flasks. Confirmation of CO_2 formation was observed through a color change, and calculations were carried out in percentage values, which for all samples fell within the range of 89 %.

The process of photocatalytic decomposition of dye in circulating conditions was carried out in a laboratory setup, the scheme of which is shown in Fig. 4.

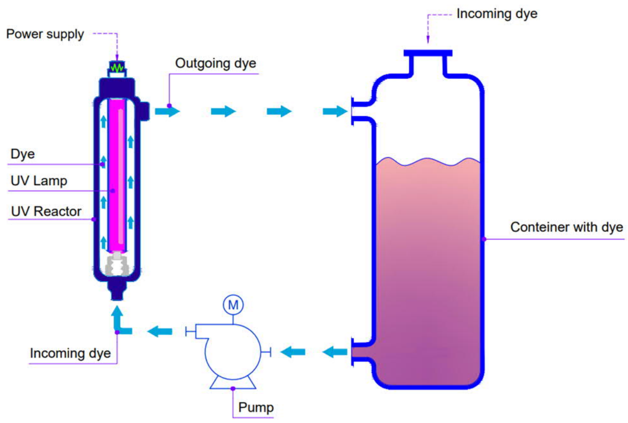

Fig. 4. *Schematic of a laboratory setup for photocatalytic decomposition of dye in circulating conditions*

The weight of ZnO/TiO_2 composite photocatalyst was pre-dispersed in a small volume of aqueous dye solution (Congo red) and the resulting suspension was added to a glass container containing 3 L aqueous dye solution and the pump was turned on to circulate it. For several minutes, the setup was operated in the dark to completely distribute the particles of the ZnO/TiO_2 composite photocatalyst in the solution volume. Then the UV lamp (λ = 254 nm and Q = 24 W) was switched on. Samples of the Congo red solution subjected to photocatalytic degradation were taken at different time intervals and filtered through a syringe membrane filter (Hutsul et al., 2022), the results of this study are shown in Fig. 5.

As can be seen from the histograms shown in Fig. 5, the efficiency of the photocatalytic degradation of the studied dye in circulating conditions strongly depends on the mass of the added composite photocatalyst, but does not depend much on the duration of the UV irradiation process.

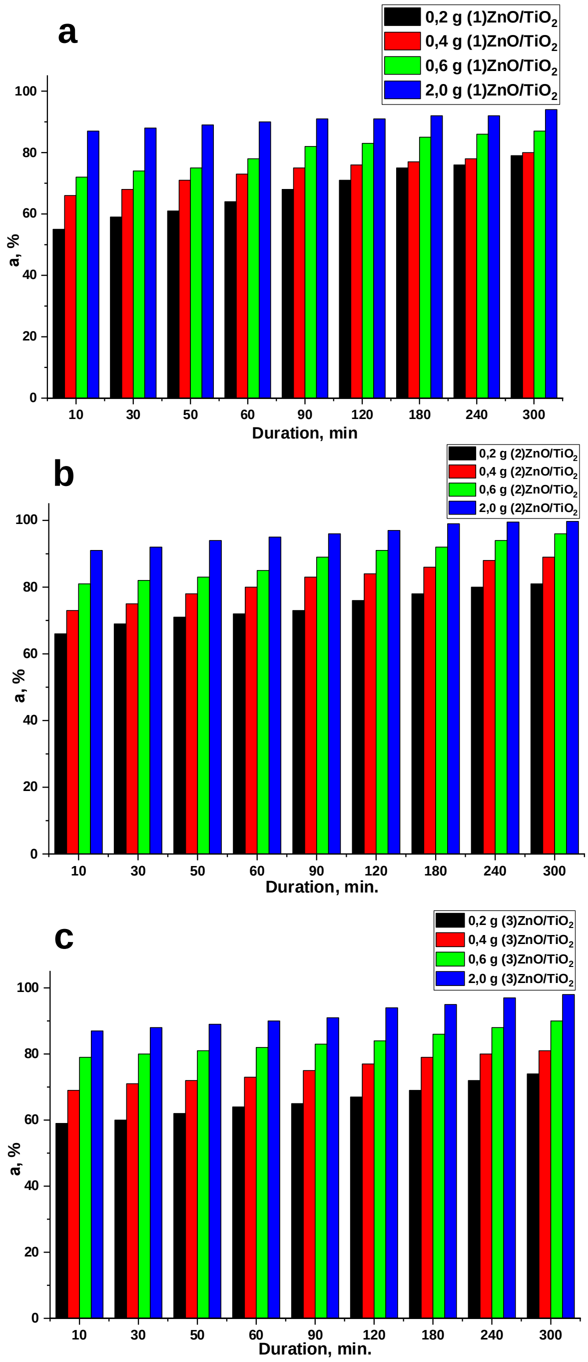

Fig. 5*. The degree of Congo red photocatalytic decomposition in circulating conditions in the presence of different weights of: a) (1)ZnO/TiO_2; b) (2)ZnO/TiO_2; c)(3)ZnO/TiO_2*

A solution of Congo red dye with an initial concentration of 25 mg/L and a volume of 3 L was decolored by 55 % in the case of the (1)ZnO/TiO_2 composite and ~60 % in the case of the other two composites after irradiation with UV light for the first 10 min and the addition of 0.2 g of the composite. During the next 290 minutes of the photocatalytic process, the solution was discolored by 79 % in the case of (1)ZnO/TiO_2 composite, and by 81 and 74 % for (2)ZnO/TiO_2 and (3)ZnO/TiO_2 composites, respectively. When using a portion of 0.4 g of the ZnO/TiO_2 composite, the dye solution was discolored by 77 % for composite (1) and (3), and by 84 % for composite (2) during two hours of the experiment, with a discoloration degree of ~70 % achieved in the first 10 min for the all three composites. The dye under study was decomposed by ~90 % after using 0.6 g of (1)ZnO/TiO_2 and (3)ZnO/TiO_2 composites. In the case of the (2)ZnO/TiO_2 composite, 96 % decomposition was achieved. In the presence of 2.0 g of ZnO/TiO_2 composites in the first 10 min of the process, the degree of decomposition in the presence of every three ZnO/TiO_2 composites reached ~90 %. With further irradiation for 300 min it increased to 94% for (1)ZnO/TiO_2) and to 98 % for (3)ZnO/TiO_2). In the case of the (2)ZnO/TiO_2 composite, the degree of decomposition of ~100 % was reached within 180 minutes.

4. Conclusions

Series of ZnO/TiO_2 composites were synthesized using the precipitation method, where ZnO is deposited on the surface of TiO_2 in the form of nano-sized layers and crystallizes in the hexagonal symmetry of wurzite type. It has been found that the optical bandgap width strongly depends on the ratio of oxides in the obtained composites, with the minimum value of 2.98 eV achieved at an equal ratio of ZnO to TiO_2.

Their photocatalytic activity was investigated under conditions approximating industrial settings, specifically on a large-volume circulation laboratory setup. The maximum effective dosage of the composite photocatalyst ZnO/TiO_2 was experimentally determined to be 2 g per 0.075 g of dye.

The experimental results demonstrated high photocatalytic activity of the synthesized ZnO/TiO_2 composites.

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ФОТОКАТАЛІТИЧНА АКТИВНІСТЬ КОМПОЗИТІВ ZnO/TiO_2 В ЦИРКУЛЯЦІЙНИХ УМОВАХ

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Розробка нових ефективних фотокаталізаторів є важливим завданням для вирішення проблем, пов'язаних з очищенням води та повітря від органічних забруднень. Композитні матеріали на основі ZnO та TiO_2 демонструють високу фотокаталітичну активність, що робить їх перспективними для даного застосування. У даній роботі представлено синтез та дослідження фотокаталітичної активності композитів ZnO/TiO_2, отриманих методом осадження проміжних гідроксидів. Синтез проводився на поверхні TiO_2 марки Evonik P25 з трьома різними масовими співвідношеннями ZnO до TiO_2: 1:3, 1:1 та 3:1. Отримані композити досліджувалися методами рентгенівської дифракції, спектроскопії для визначення оптичної ширини забороненої зони та піддавали фотокаталітичному розкладанню в циркуляційних умовах. За допомогою рентгенівської дифракції було підтверджено, що ZnO кристалізується у вюрцитовій фазі гексагональної сингонії, а TiO_2 міститься у вигляді двох модифікацій: анатазу та рутилу. Досліджено вплив масового співвідношення ZnO до TiO_2 на оптичну ширину забороненої зони. Використовуючи алгоритм Кубелки-Мунка, було визначено оптичну ширину забороненої зони композитів ZnO/TiO_2. Для композитів (1)ZnO/TiO_2 та (3)ZnO/TiO_2 ширина забороненої зони становила 3,22 еВ, а найменше значення (2,99 еВ) було отримано для композиту з рівним співвідношенням ZnO до TiO_2 - (2)ZnO/TiO_2. Фотокаталітична активність композитів ZnO/TiO_2 досліджувалась в циркуляційних умовах з барвником конго червоним в присутності чотирьох різних наважок композитів: 0,2; 0,4; 0,6 та 2 г. Максимальна ефективність фотокаталітичного розкладання барвника спостерігалась для композиту з рівним співвідношенням ZnO до TiO_2 при дозуванні 2 г композиту на 0,075 г барвника. Синтезовані композити ZnO/TiO_2 демонструють високу фотокаталітичну активність, що робить їх перспективними матеріалами для очищення води та повітря від органічних забруднень. Оптимальне масове співвідношення ZnO до TiO_2 для фотокаталітичного розкладання барвника конго червоного становить 1:1.

Ключові слова: *барвник, композит, конго червоний, титан (IV) оксид, фотокаталіз, цинк (II) оксид.*