PROSPECTS FOR THE APPLICATION OF ZINC OXIDE SYNTHESIZED BY HYDROTHERMAL METHOD

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Nanostructured oxides with photocatalytic properties are promising materials for use in such directions as water treatment from organic pollutants and production of self-cleaning coatings. Purification is carried out due to the gradual degradation of organic pollutants under the action of radicals formed on the surface of the photocatalyst. Zinc oxide is one of the effective photocatalytic materials. The article investigates the effect of synthesis conditions on the properties of zinc oxide. Zinc hydroxide was precipitated from a zinc acetate solution by a sodium hydroxide solution. In order to reduce the size of the crystallites, before precipitation the zinc acetate solution was mixed with a guar gum solution. The resulting suspension was heat-treated in a sealed container without air access at a temperature of 110 °C. After that, the material was filtered, rinsed and heated at temperatures of 150 °C and 500 °C. The composition of the samples was studied by X-ray diffraction. It was found that the use of the hydrothermal method with heat treatment at a temperature of 150 °C allows obtaining a mixture of hexagonal crystalline modification of zinc oxide, cubic crystalline modification of zinc oxide and smithsonite. After heating at a temperature of 500 °C, only hexagonal modification of zinc oxide was obtained. The photocatalytic efficiency was investigated using solutions of congo red and crystal violet. It was determined that the presence of cubic modification of zinc oxide in the material increases the efficiency of the photocatalyst. This makes the material a promising photocatalyst both in the context of water treatment and in applying of self-cleaning coatings.

Keywords: *metal oxide, nanostructured oxides, photocatalysis, self-cleaning coatings, water treatment*

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1. Introduction

The annual world wastewater discharge is about 1500 km^3 (Kumar et al., 2022). Organic pollutants, including components of medicines and cosmetics, household chemicals, etc., are often present in wastewater. Such pollutants are often resistant to destruction by microorganisms at the stage of biological wastewater treatment, have a negative impact on aquatic organisms and tend to accumulate in ecosystems. In recent decades, a multitude of substances have been detected in the aquatic environment, either entirely anthropogenic or naturally occurring compounds that were previously found in nature at very low concentrations (Kumar et al., 2022).

Although most surfactants are biodegradable and low-toxic, they can cause significant harm by increasing the ability of other toxic components to penetrate the body (Ivanković & Hrenović, 2010).

Pharmaceuticals and personal care products have toxic effects on aquatic organisms (fish, invertebrates, etc.), plants, animals and humans. Pollutants of this type often exhibit genotoxic and carcinogenic effects, disruption of endocrine, reproductive and behavioral systems, as well as many other effects (Wydro et al., 2023).

Endocrine disruptors are often enter to wastewater as components of pharmaceuticals and personal care products, industrial solvents/lubricants, plastics (bisphenol A, phthalates, etc.), and many others (Kumar et al., 2022). Very often, these pollutants remain in the water not only after wastewater treatment, but even after drinking water treatment plants. The content of pharmaceuticals and personal care products in tap water is in the range of 0.16–32.5 ng/L (Wydro et al., 2023). Endocrine disruptors affect the hormonal balance through a variety of mechanisms, including disrupting hormone production, mimicking hormone action, affecting the development of hormone receptors, acting as hormone antagonists, and altering hormone binding (Kumar et al., 2022). Endocrine disruptors are physiologically active even at very low concentrations (a few nanograms per liter) and can accumulate in the environment, especially in water (Kumar et al., 2022).

In addition, the entry of small concentrations of antibiotics into ecosystems leads to the development of antibiotic resistance in various types of microorganisms, including conditionally pathogenic and pathogenic (Litynska et al., 2021).

Therefore, complete removal of such pollutants is extremely necessary during wastewater treatment, but this is unattainable for the technologies currently used in Ukraine at wastewater treatment plants (Litynska, 2023). This is causing growing concern about the state of ecosystems.

Photocatalytic wastewater treatment technology was developed over 30 years ago, but it is still rarely used in industry (Mei et al., 2023). But the development of visible light photocatalysts, including those based on zinc oxide, for wastewater treatment has received significant attention due to their ability to operate under low energy consumption conditions and the absence of the generation of significant amounts of waste that require disposal (Zheng et al., 2022).

Another problem associated with pollution is the formation of layers of dirt on various types of household and technological surfaces, which mainly consist of fats, microorganisms, dust, etc. Transparent and self-cleaning protective coatings are widely used in the manufacture of car windows, camera lenses, various electronic gadgets, etc. (Wu et al., 2023). The development of self-cleaning coatings will allow avoiding the deposition of organic components on surfaces due to not only superhydrophobic or superhydrophilic properties, but also the destruction of organic molecules and the disinfecting effect on microorganisms.

In addition, in the case of glass surfaces, such coatings can also provide them with anti-reflective properties. In the case of solar panels, self-cleaning anti-reflective coatings allow for increased electricity generation without changing the panel area (Arabatzis et al., 2017). According to (Wijesingha et al., 2024), spin-coated ZnO is an effective anti-reflective coating for CdS/CdTe solar cell devices and demonstretes an average short-circuit current density increasing of 6.8% and improvement in power conversion efficiency of 9.3%.

Therefore, oxide photocatalysts, including zinc oxide, are highly promising materials in several areas:

1) wastewater treatment of organic pollutants that are difficult to remove by traditional biological or adsorption methods;

2) creation of self-cleaning protective coatings;

3) creation of anti-reflective coatings.

2. Materials and Methods

Photocatalyst synthesis. Figures 1 and 2 demonstrate the stages of photocatalyst synthesis. 10 g of zinc acetate was dissolved in 50 mL of distilled water. This solution was mixed by a magnetic stirrer with 50 mL of guar gum solution with concentration 2 g/L. Zinc hydroxide was precipitated from a zinc acetate solution by a sodium hydroxide solution. Addition of sodium hydroxide solution with concentration 0.1 mol/L was gradually carried out until pH 7 was reached.

The resulting suspension was heat-treated in a sealed container without air access at a temperature of 110 °C during 4 hours.

After cooling, the mixture was filtered under vacuum through a cellulose acetate filter with a pore size of 0.45 μm and rinsed by distilled water. Obtained material was dried at 150°C and powdered in a mortar.

Half of the powder was calcined in a muffle furnace at 500°C for 1 hour.

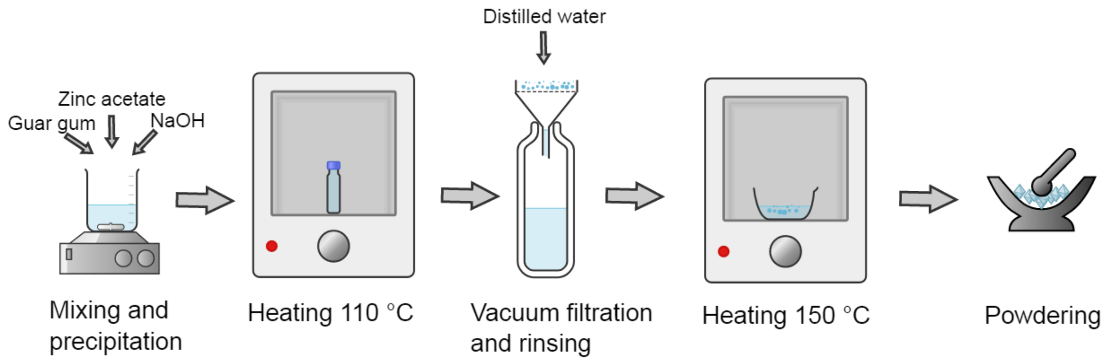

Fig. 1. *Stages of synthesis of H-150 photocatalyst*

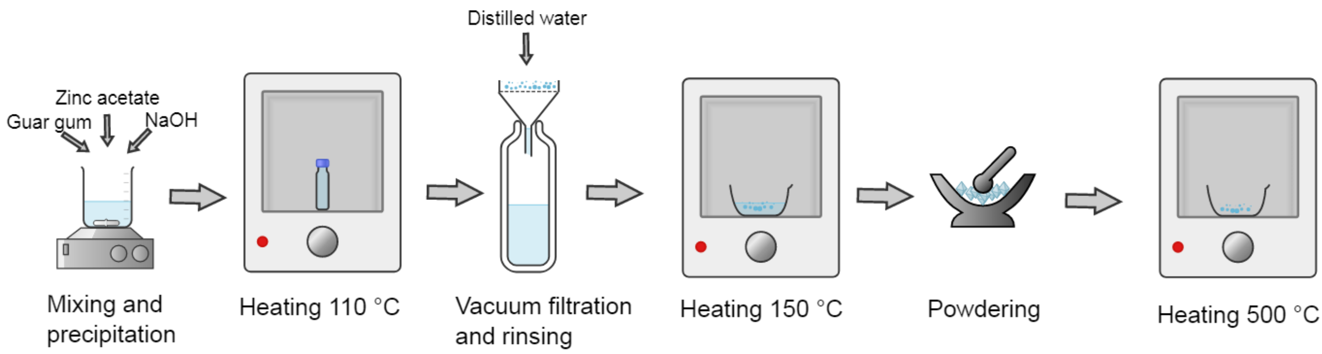

Fig. 2. *Stages of synthesis of H-500 photocatalyst*

Composition research. X-ray diffraction was carried out using Rigaku Ultima IV (Japan) with CuKa radiation (20-60 kV, 2-60 μA). The obtained diffractograms were analyzed by automatic comparison with the cards of pure chemical compounds available in the database. Identification was carried out based on the results of the coincidence of the 2θ angles of the diffractogram of the sample and the card.

Photocatalityc research. The photocatalytic efficiency was tested using solutions of crystal violet (Fig. 3) and congo red (Fig. 4) with a concentration of 25 mg/L.

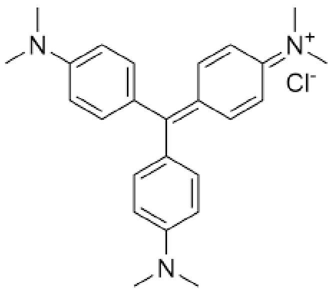

Fig 3. *Chemical structure of crystal violet*

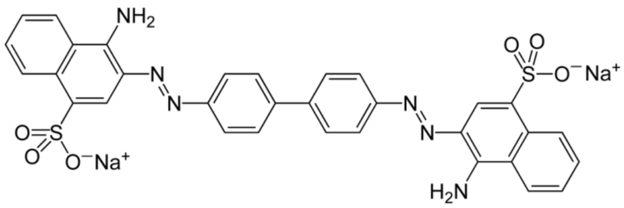

Fig 4. *Chemical structure of congo red*

A portion of the photocatalyst (0.02 g) and 20 ml of the dye solution were mixed in a glass container. The resulting suspensions were placed under a UV lamp for photocatalytic destruction of the dye with constant stirring using a magnetic stirrer. The durations of photocatalysis were 15 and 30 min. Separation of the photocatalyst from the solution using a syringe filter with a pore diameter of 0.22 μm.

Spectra of the filtrates were obtained using a 1 cm quartz cuvette on the spectrophotometer Shimadzu UV-2600i (Japan).

3. Results and Discussion

According to X-ray diffraction data, the crystallite size of H-150 was 8.1-8.8 nm, and for H-500 crystallite size was 15.0 nm. That is, with increasing heat treatment temperature, the size of the crystallites increased.

Figure 5 shows the diffractograms of the samples. Material H-150 consisted of hexagonal crystalline modification of ZnO, cubic crystalline modification of ZnO and smithsonite ($ZnCO_3$). While sample H-500 contained only one phase (hexagonal crystalline modification of ZnO).

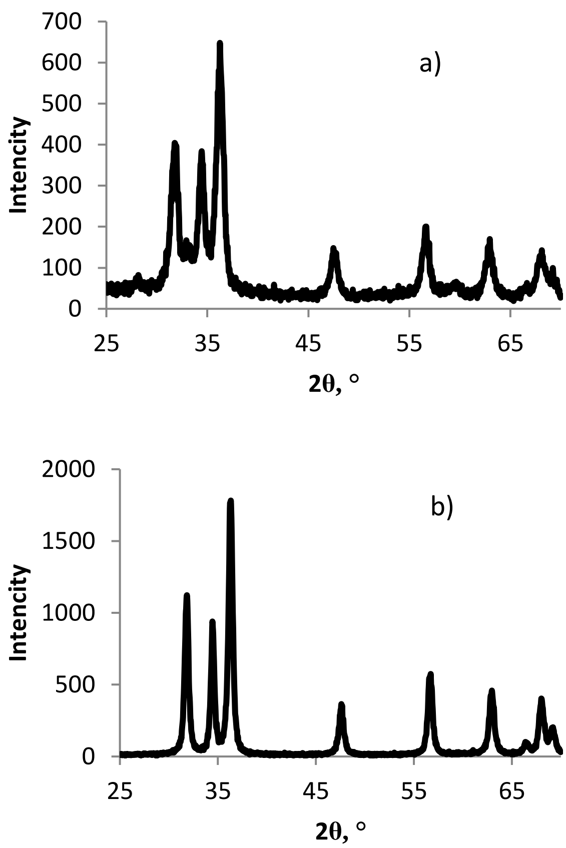

Fig 5. *X-ray diffractograms: a) H-150; b) H-500*

Therefore, with increasing heat treatment temperature, the cubic crystalline modification changes to the hexagonal one, as well as the decomposition of smithsonite and amorphous impurities to obtain the hexagonal crystalline modification of zinc oxide.

The photocatalytic efficiency was investigated using solutions of congo red and crystal violet.

Figures 6 and 7 demonstrate the effect of heat treatment conditions on the photocatalyst efficiency towards congo red.

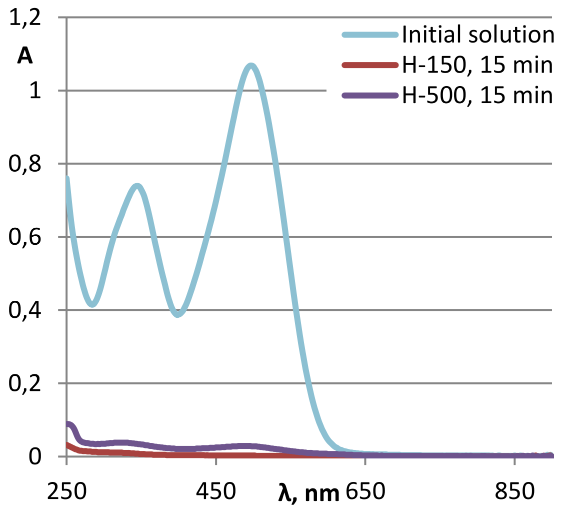

Fig 6. *Effect of synthesis conditions on the efficiency of congo red destruction during of 15 min*

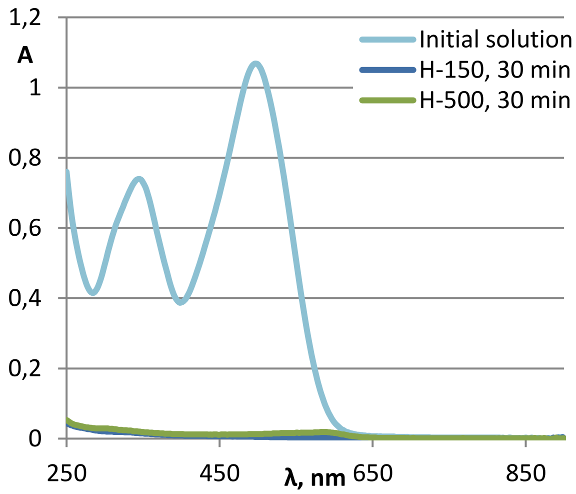

Fig 7. *Effect of synthesis conditions on the efficiency of congo red destruction during of 30 min*

Both samples demonstrated high efficiency in removing Congo red even in 15 min, but H-150 is slightly more efficient than H-500.

In the case of crystal violet (Fig 8 and 9), a similar trend was observed and H-150 was more effective than H-500.

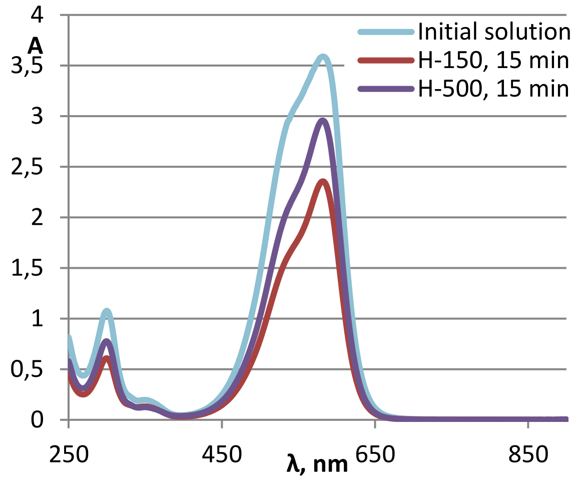

Fig 8. *Effect of synthesis conditions on the efficiency of crystal violet destruction with a photocatalysis duration of 15 min*

Although after 30 min of photocatalysis (Fig 9), the removal efficiency of crystal violet was higher than after 15 min (Fig. 8), it was still far from complete destruction.

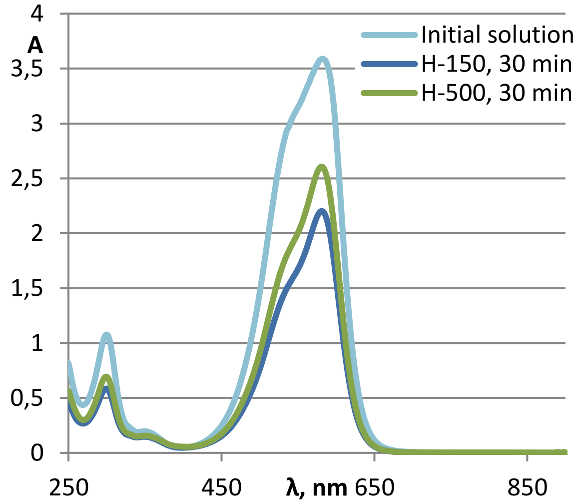

Fig 9. *Effect of synthesis conditions on the efficiency of crystal violet destruction with a photocatalysis duration of 30 min*

It was determined that the presence of cubic modification of zinc oxide in the material increased the efficiency of the photocatalyst. Another possible factor is the size of the crystallites, as in H-150 it is smaller than in H-500. This made the material a promising photocatalyst both in the context of water treatment and in applying of self-cleaning coatings.

4. Conclusions

The synthesized photocatalytic materials demonstrated high efficiency in the destruction of congo red but lower efficiency towards crystal violet, which is explained by the difference in the structure of these dyes. The crystal violet molecule is more resistant to destruction. Thus, a longer duration of photocatalysis was required for complete destruction of crystal violet.

To achieve high efficiency, the heat treatment temperature should not exceed 150 °C, as this will lead to an increase in crystallites and the transition of the cubic crystalline modification of zinc oxide to the less efficient hexagonal modification.

Therefore, zinc oxide synthesized by the hydrothermal method is a promising material for wastewater treatment and the creation of self-cleaning coatings, including for solar panels.

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ПЕРСПЕКТИВИ ЗАСТОСУВАННЯ СИНТЕЗОВАНОГО ГІДРОТЕРМАЛЬНИМ МЕТОДОМ ЦИНК ОКСИДУ

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Наноструктурні оксиди з фотокаталітичними властивостями є перспективними матеріалами для використання в таких галузях як очищення вод від органічних полютантів та нанесення самоочисних покриттів. Очищення здійснюється за рахунок поступової деградації органічних полютантів під дією радикалів, утворених на поверхні фотокаталізатору. Цинк оксид є одним з ефективних фотокаталітичних матеріалів. У статті досліджено вплив умов синтезу на властивості цинк оксиду. Цинк гідроксид було осаджено з розчину цинк ацетату розчином натрію гідроксиду. З метою зменшення розмірів кристалітів перед осадженням розчин цинк ацетату було змішано з розчином гуарової камеді. Одержану суспензію було теромооброблено в герметичній ємності без доступу повітря за температури 110 °С. Після цього матеріал було відфільтровано, промито дистильованою водою та термооброблено за температур 150 та 500 °С. Склад зразків було досліджено методом рентгенівської дифракції. Встановлено, що використання гідротермального методу з термообробкою за температури 150 °С дозволяє одержати суміш гексагональної кристалічної модифікації цинк оксиду, кубічної кристалічної модифікації цинк оксиду та смітсоніту. Після термообробки за температури 500 °С одержується лише гексагональна модифікація цинк оксиду. Фотокаталітична ефективність була досліджена з використанням розчинів конго червоного та кристалічного фіолетового. Визначено, що наявність у складі матеріалу кубічної модифікації цинк оксиду підвищує ефективність фотокаталізатору. Це робить матеріал перспективним фотокаталізатором як у контексті водоочищення, так і контексті нанесення самоочисних покриттів.

Ключові слова: *наноструктурні оксиди, оксид металу, очищення води, самоочисні покриття, фотокаталіз*