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Preparation of ZnO Nanoparticles by Arc-Plasma Discharge Method

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In this paper, ZnO nanoparticles are synthesized by arc-plasma discharge method. The obtained nanoparticles are characterized using IR spectroscopy and XRD method. The IR results show the absorption band of Zn–O at 455 cm^{-1} , while the powder XRD pattern confirms the hexagonal wurtzite structure with particles' size of 48.06 nm. ZnO nanoparticles are used as catalyst for degradation of methylene blue, using hydrogen peroxide as an oxidant. The catalytic activity is estimated.

У цій роботі наночастинки ZnO було синтезовано методом дугового плазмового розряду. Одержані наночастинки було охарактеризовано за допомогою ІЧ-спектроскопії та рентгенівської дифрактометрії. ІЧ-результати показують смугу вбирання Zn–O в околі 455 cm^{-1} , тоді як порошкова рентгенівська дифрактограма підтвердила вюртцитову гексагональну структуру з розміром частинок у 48,06 нм. Наночастинки ZnO було використано як каталізатор для деградації метиленового синього з застосуванням перекису Гідрогену як окиснювача. Було оцінено каталітичну активність.

Key words: ZnO nanoparticles, arc-plasma discharge, catalytic activity, methylene blue degradation.

Ключові слова: наночастинки ZnO, дуговий плазмовий розряд, каталітична активність, деградація метиленового синього.

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1. INTRODUCTION

Recently, zinc oxide has attracted much attention within the scien-

tific community as a future material due to its application in UV light-emitters, varistors, gas sensors, surface acoustic wave devices, piezoelectric transducers and as a window material for displays and solar cells [1, 2]. Several methods including chemical vapour deposition [3], sol-gel processing [4], magnetron sputtering [5], vacuum evaporation [6], pulse laser ablation [7] and electrical arc discharge in vacuum conditions under argon and oxygen atmosphere [8, 9] have been used to prepare ZnO nanoparticles (NPs). Among physical and chemical methods, electrical arc discharge in liquid is an attractive method because of simplicity of apparatus building, low impurity introduction, no need for complicated vacuum equipments, high-throughput and cost-effective procedure to generate a high yield of nanoparticles. The simplicity of this method also allows scaling up for mass production. In recent years, a few papers have been published regarding synthesis of NPs such as CuO and Cu₂O [10], Ag [11], Au [12] and WO₃ [13] by arc discharge in liquids.

The aim of this study is to prepare ZnO NPs, using arc-plasma discharge method, and examine the catalytic properties of the ZnO nanocomposites for methylene blue (MB) oxidation.

2. EXPERIMENTAL

2.1. Preparation of ZnO Nanoparticles

The preparation system consists of two main parts, a high current DC power supply and a reactor including anode, cathode and micrometer, which moves the anode towards the cathode. In this work, 5–15 A current was applied between two zinc electrodes. The voltage was dropped to about 2–3.5 V during the arc, while the current was fixed to desired amount. Both anode and cathode were wires shaped 1 mm in diameter and 99.99% purity. Initially, we bring the two electrodes into touch leading to a small contact cross-section and, thus, to a high current density. As a result, zinc is ablated from the anode and then condensed in water. These chemicals were utilized in the synthesis of the samples using distilled water.

2.2. Characterization

The crystallite structure and size of the synthesized nanoparticle samples were calculated using an x-ray powder diffractometer (XRD) Philips-PW-1840 with a CuK_α radiation of 1.5418 Å in a 2θ range of 20–80°, while the analysis of the chemical composition was carried out using Fourier transform infrared spectroscopy (FT-IR model: Jasco Spectrum 4100 FT-IR).

2.3. Catalytic Degradation Experiments

The degradation experiments were performed in the glass bottle of 250 mL containing 100 mL of MB solution. In details, a certain amount of catalyst (0.1 gr.) was added to the MB solution of 64 mg/L under stirring for 1 h in the dark. 10 mL of H₂O₂ solution 30% v/v was added.

The ultraviolet-visible (UV-Vis) absorbance of supernatant was determined at wavelength of 665 nm (for MB). The catalytic activity (*Ca*, %) of MB was calculated as

$$Ca = \frac{A_0 - A_e}{A_0} \cdot 100\%, \quad (1)$$

where A_0 and A_e are the initial and equilibrium absorptions of MB, respectively.

3. RESULTS AND DISCUSSION

3.1. FT-IR Spectroscopy

The FT-IR spectra of ZnO nanoparticles are presented in Fig. 1. All FT-IR spectra results were observed in the range of 400–4000 cm⁻¹ at room temperature and are presented in Table. The most important absorption bands in the spectra are at 455 cm⁻¹, 493 cm⁻¹, which belong to Zn–O, Zn–O–Zn, respectively.

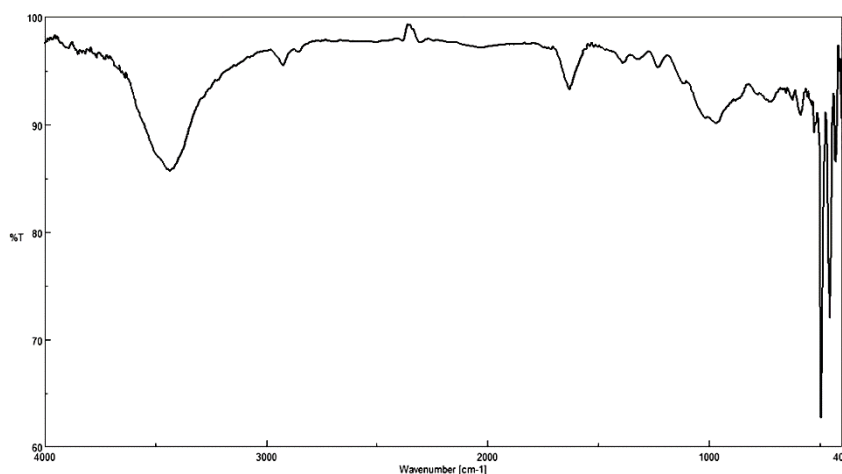
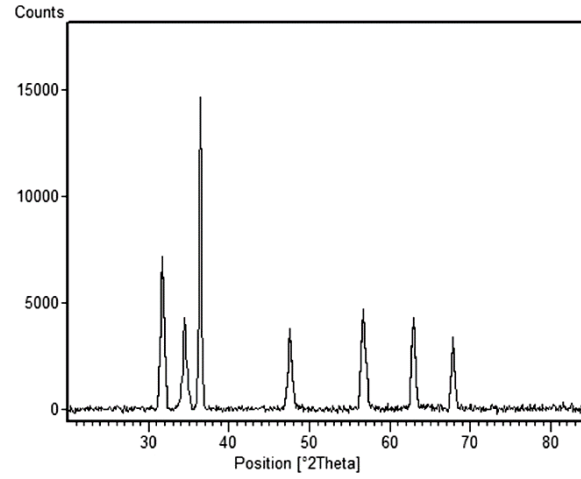


Fig. 1. FT-IR spectra of synthesized ZnO NPs.

TABLE. The absorption-bands wave number of the FT-IR spectra of ZnO NPs.

Wave number, cm^{-1}	Bond type
3436	O-H (stretching)
1631	H-O-H (bending)
493	Zn-O-Zn (stretching)
455	Zn-O (stretching)

**Fig. 2.** XRD patterns of ZnO NPs.

3.2. XRD analysis

The XRD pattern for the prepared ZnO nanoparticles was shown in Fig. 2. The phase of the samples was a hexagonal structure. The obtained spectrum has the first ZnO main peaks at $2\theta = 31.7^\circ$, 34.4° , 36.2° , 47.5° , 56.6° , 62.8° and 67.9° . The calculated unit cell parameters ($a = b = 3.245 \text{ \AA}$, $c = 5.191 \text{ \AA}$) are in a good agreement with the values reported in the JCPDS file # 96-230-0113.

Figure 3 shows the lattice unit cell of prepared metal ferrite.

The average crystallite size was calculated using Scherrer's equation, which is expressed as follows [14]:

$$D = \frac{K\lambda}{\beta \cos \theta}, \quad (2)$$

where K is the Scherrer's constant ($K = 0.89$, if assuming spherical particles), λ is the x-ray wavelength, β is the peak full-width at half maximum ($FWHM$), and θ is the Bragg's diffraction angle. The av-

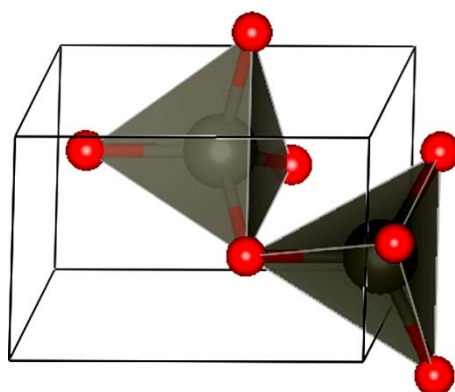


Fig. 3. Lattice cell of ZnO NPs.

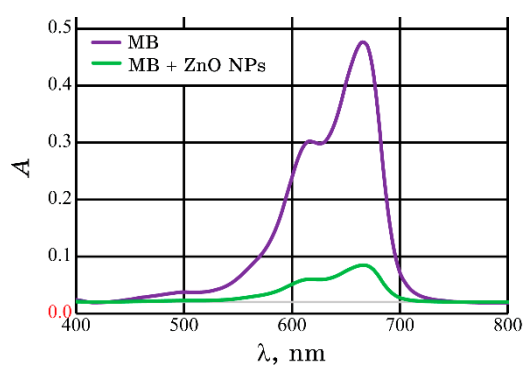


Fig. 4. UV–Vis spectra of MB with and without ZnO NPs.

erage crystallite size was of 48.06 nm.

3.3 The Catalytic Activity

The catalytic activity of ZnO NPs prepared by the arc-plasma discharge method was studied through the methylene blue oxidation reaction using hydrogen peroxide. Figure 4 shows the absorption spectra of methylene blue with and without ZnO after 60 min of reaction time. The catalytic activity was calculated by means of Eq. (1). As can be seen from the previous results, the catalytic activity reached to 85.8%.

4. CONCLUSION

ZnO NPs were successfully synthesized using arc-plasma discharge

method. The resulting compounds were characterized using XRD techniques, which confirmed the size of nanoparticles, and IR spectroscopy. The catalytic activity was studied, and it was found that the ZnO NPs have a high catalytic activity.

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