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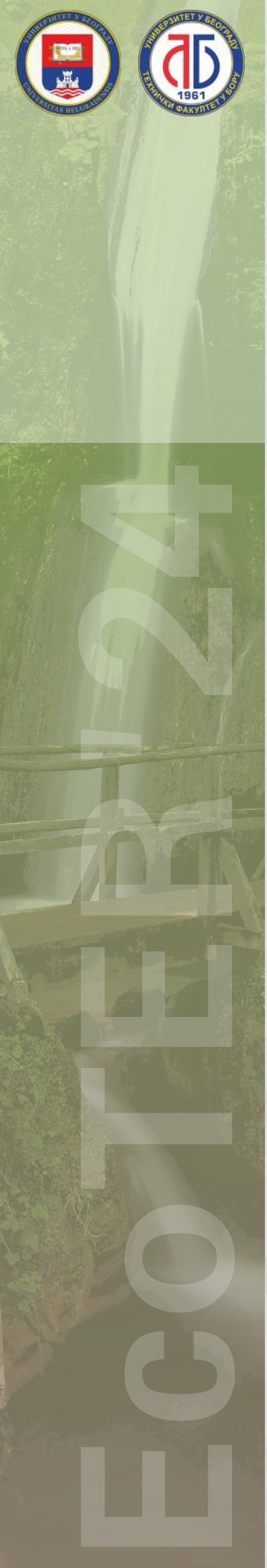

31st International conference

Ecological Truth & Environmental Research

Editor
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PROCEEDINGS

Hotel Sunce, Sokobanja, Serbia
18–21 June 2024

PROCEEDINGS

31st INTERNATIONAL CONFERENCE

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PREFACE

The 31st international conference Ecological Truth & Environmental Research – EcoTER'24 focuses on showing the latest research findings and innovations in the field of ecology, environmental protection and sustainable development. The conference will be held in Sokobanja (Serbia) in hotel Sunce in the period of 18–21 June 2024.

The aim of the conference is to connect the experts in various fields in order to transform attitudes and behaviors in everyday practices, as well as in the industry and economy sector which is essential for achieving the desired changes that our society must undergo.

The 31st international conference Ecological Truth & Environmental Research – EcoTER'24 is organized by the University of Belgrade, Technical Faculty in Bor, and co-organized by the University of Banja Luka, Faculty of Technology; the University of Montenegro, Faculty of Metallurgy and Technology – Podgorica; the University of Zagreb, Faculty of Metallurgy – Sisak; the University of Pristina, Faculty of Technical Sciences – Kosovska Mitrovica and the Society of Young Researchers – Bor.

These Proceedings encompass 119 papers from the authors coming from the universities, research institutes and industries in 15 countries: Brazil, Norway, USA, Spain, Austria, Libya, Italy, Israel, Slovenia, Croatia, Romania, Bulgaria, Montenegro, Bosnia and Herzegovina, North Macedonia, and Serbia. It is a great honor and pleasure to cordially wish a warm welcome to all the participants of the conference.

As a part of this year's conference, the 6th Student Section – EcoTERS'24 will be held. We appreciate the contribution of the students and their mentors who have also participated in the conference and hope that students will continue to explore and to be curious, since education is a never-ending process, and knowledge is continuously growing.

The organization of the EcoTER'24 conference has been financially supported by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia.

The support of the Donors and their willingness and ability to cooperate has been of great importance for the success of the EcoTER'24 conference. The organizing committee would like to extend their appreciation and gratitude to the Platinum donors of the conference – Serbia ZiJin Copper doo Bor and HBIS SERBIA, to the Gold donor of the conference – Elixir Group, as well as to the Silver donor of the conference – Serbian Chamber of Engineers.

We would like to express our sincere appreciation to all the authors who have contributed to the Proceedings. We would also like to express our gratitude to the members of the scientific, organizing and honorary committees, reviewers, speakers, chairpersons and all the conference participants for their support of the EcoTER'24. Sincere thanks go to all the people who have contributed to the successful organization of the EcoTER'24.

Prof. Snežana Šerbula,

President of the scientific and organizing committee

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PHOTOCATALYTIC PROPERTIES OF C-ZnO NANOPARTICLES SYNTHESIZED *via* MECHANOCHEMICAL METHOD

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Abstract

Escalating water pollution necessitates the development of efficient and sustainable photocatalytic materials for the degradation of organic pollutants. In this work, the photocatalytic properties of carbon-doped zinc oxide (C-ZnO) nanoparticles synthesized by a novel mechanochemical method are investigated. The unique approach of doping ZnO nanoparticles with carbon not only increases the absorption of visible light but also improves the efficiency of charge separation, leading to a significant increase in photocatalytic activity. Experimental analytical methods such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV-Vis spectroscopy are used to elucidate the structural, morphological, and photocatalytic properties of the synthesized C-ZnO nanoparticles. The photocatalytic performance of C-ZnO nanoparticles is evaluated by their ability to degrade methyl orange at a concentration of 5 ppm under UV light irradiation with and without mixing the solution for 60 minutes in the dark before exposure to UV light. The influence of the photocatalysis time on the degradation efficiency was investigated. The results show that the photocatalysis time has a strong influence on the degradation efficiency.

Keywords: photocatalysis, nanomaterials, doping, mechanochemical, synthesis.

INTRODUCTION

Zinc oxide nanoparticles (ZnO-NP) have attracted considerable attention in the field of photocatalysis due to their excellent optical properties, high electron mobility, and environmental friendliness. As a wide bandgap semiconductor (approximately 3.37 eV), ZnO inherently has strong absorption of ultraviolet (UV) light, but limited activity in visible light, which makes up the majority of the solar spectrum. This limitation has led to extensive research into the modification of ZnO to harness the broader spectrum of solar energy for photocatalytic applications, including the degradation of organic pollutants [1,2].

Among various strategies to extend the photocatalytic efficiency of ZnO NPs into the visible light range, doping with atoms of different elements has been identified as a particularly effective method. Doping introduces impurity levels within the bandgap of ZnO, which can facilitate the absorption of visible light by narrowing the bandgap or creating

mid-gap states that act as stepping stones for electron transition from the valence to the conduction band. In particular, carbon doping has proven to be a promising approach, as carbon can introduce p-type conductivity into ZnO, thereby improving charge separation and reducing the electron-hole recombination rate [3,4].

Carbon-doped ZnO nanoparticles (C-ZnO NP) exhibit significantly improved photocatalytic activity under visible light compared to their undoped counterparts. The incorporation of carbon into the ZnO lattice or the ZnO surface can lead to the formation of C-O-Zn bonds, which are believed to contribute to the generation of active sites for photocatalytic reactions. Furthermore, carbon doping can enhance the adsorption of pollutants on the catalyst surface, facilitating their subsequent degradation [4].

Recent studies have demonstrated the effectiveness of C-ZnO NPs in degrading various organic pollutants, under UV and/or visible light irradiation. For instance, the carbon doping of ZnO using natural precursors such as glucose or other similar chemicals offers an environmentally and cost-effective way to synthesize photocatalysts with enhanced activity and stability [5].

Despite the progress in the synthesis of C-ZnO NP, the challenge is to optimize the doping level, understand the exact mechanisms of photocatalytic enhancement and increase production for practical applications.

MATERIALS AND METHODS

Sample preparation

ZnO NPs, both undoped and carbon-doped, were produced using a mechanochemical method followed by a calcination process. The synthesis of undoped and carbon-doped ZnO nanoparticles involved mixing zinc acetate dihydrate (Sigma-Aldrich) with oxalic acid dihydrate (Sigma-Aldrich) and grinding them in an agate mortar for 10 minutes, resulting in a gel composed of zinc oxalate dihydrate and acetic acid. For the synthesis of C-ZnO NP, glucose (Merck) was additionally incorporated into the gel, with the grinding extended for another 10 minutes to form the precursor. The final undoped and C-ZnO NP were then produced by calcining this mixture at 500°C for 3 hours.

Characterization methods

SEM photograph was obtained via QUANTA FEG 250 SEM microscope. The X-ray diffraction (XRD) patterns of the C-ZnO NP were recorded using an X'Pert³ Powder X-ray diffractometer for a range of 2θ from 20–90°. The photocatalytic activity of synthesized nanomaterial was accessed via UV/VIS spectrometry.

RESULTS AND DISCUSSION

SEM photograph is presented in Figure 1. The SEM image shows a closer look at the nanorods, which differ in shape, size, and surface roughness, which can be related to the growth conditions and carbon concentration during the synthesis process [6,7]. The diameter and length of the smallest nanorods are about 30 nm.

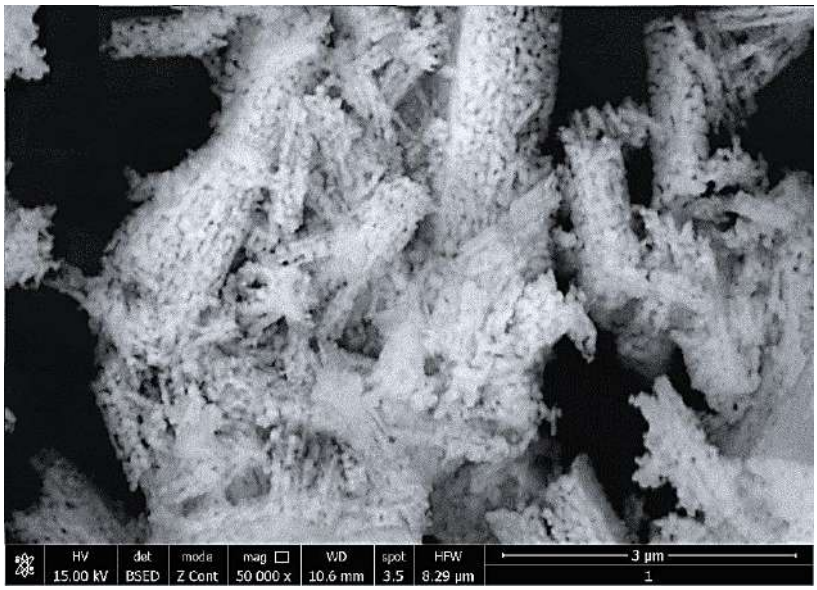

Figure 1 *SEM image of synthesized carbon-doped ZnO NP*

The XRD pattern of the synthesized doped nanoparticles is presented in Figure 2. The X-ray diffraction pattern (XRD) image shows the crystalline structure of zinc oxide doped with 5% carbon compared to undoped ZnO as defined by the Joint Committee on Powder Diffraction Standards (JCPDS) [8]. The presence of distinct peaks at angles of 33.48°, 36.10°, 37.92° and 58.07°, which align with the undoped ZnO (JCPDS no. 36–1451), confirms the hexagonal wurtzite structure of ZnO without secondary phases. For the C-ZnO NP, there is an observed shift of the (002) peak towards higher angles compared to the ZnO NP JCPDS data. This indicates a decrease in the lattice constant "c" due to carbon doping, which is expected due to the incorporation of smaller carbon ions into the oxygen sites of the ZnO lattice [9].

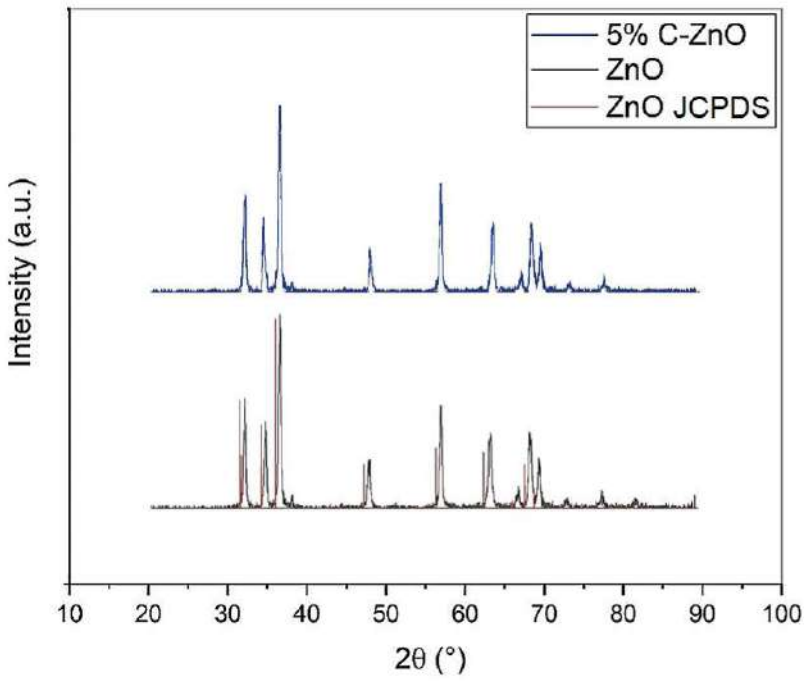

Figure 2 *XRD pattern of synthesized samples*

Based on XRD analysis and calculations, data was obtained on crystallite parameters α and c), c/α ratio, the crystallite size (D), interplanar distance (d), unit cell volume (V), Atomic packing fraction (APF), dislocation density (δ), atomic displacements (u), micro-strain (ε), number of unit cells contained in a grain (n), stacking fault (α*), stress (σ), Young's modulus (Y) and lattice volume (U)**.** Data is presented in Table 1.

Table 1 *Structural parameters of the analyzed NP samples*

Sample	α (Å)	c (Å)	c/α	D (nm)	d (nm)	V (Å^3)	APF (%)	δ·10^{-3} (nm^{-2})
ZnO	3.24	5.2	1.47	68.05	0.27	55.92	0.59	0.21
5%C-ZnO	3.09	4.97	1.61	25.32	0.25	41.06	0.53	1.56
Sample	**u**	**ε·10^{-3}**	**n·10^6**	**α***	**σ·10^7 (Pa)**	**Y·10^{10} (Pa)**		**U·10^4 (J/m^3)**
ZnO	0.4	1.2	31.6	0.0012	4.37	3.75		2.55
5%C-ZnO	0.38	1.2	2.19	0.0011	4.37	3.75		2.55

Table 1 presents the structural parameters of ZnO and 5% carbon-doped ZnO (5%C-ZnO) nanoparticle samples. Key differences between the samples highlight the impact of carbon doping on the structural and physical properties. Notably, doping reduces the crystallite size (D) from 68.05 nm in pure ZnO to 25.32 nm in 5%C-ZnO, which can enhance surface area and reactivity. Moreover, the unit cell volume (V) decreases from 55.92 Å^3 to 41.06 Å^3, suggesting a more compact structure. The higher dislocation density (δ) in 5%C-ZnO ($1.56\cdot10^{-3}$ nm^{-2}) compared to pure ZnO ($0.21\cdot10^{-3}$ nm^{-2}) indicates increased lattice imperfections, which can facilitate charge separation and improve photocatalytic activity [10,11].

To gain insight into photocatalytic properties, model compound degradation experiments were conducted with the previously prepared 5 ppm methyl orange stock solution. Experiments were conducted with and without mixing in the dark for 60 minutes before exposing to UV light irradiation (λ=395 nm), since it has been reported that in this step equilibrium for the adsorption and desorption of dye molecules is achieved. For the collected data, a pseudo-first-order model was employed and is expressed as [12,13]:

$$\ln\left(\frac{C_0}{C_t}\right) = kt \tag{1}$$

Where C_0 and C_t represent model compound concentration values at zeroth and time "t", while "k" represents the reaction rate constant. Figure 3a shows the degradation efficiency of synthesized nanoparticles, with and without mixing step in the dark for 60 minutes. The plot of $\ln(C_0/C_t)$ vs irradiation time "t" is depicted in Figure 3b and a linear relationship is observed. Reaction rate constants are evaluated from the slopes of $\ln(C_0/C_t)$ vs time plot.

The degradation efficiency of undoped and carbon-doped ZnO NPs can reach 78.93% and 89.10% in 60 minutes, which is higher than the degradation efficiency values determined for experiments without mixing in the dark. Figure 3b shows the k values for degradation

experiments. The rate constants (k) are highest for the 5%C-ZnO 500°C sample in an experiment with (k_4=0.0360 min^{-1}), and without mixing in the dark for 60 minutes before the (k_3=0.0316 min^{-1}). The pure ZnO samples have lower rate constants (k_1=0.0235 min^{-1} and k_2=0.0243 min^{-1} for experiments with and without mixing in the dark, respectively). This indicates faster degradation kinetics for the carbon-doped samples, corroborating their superior photocatalytic activity.

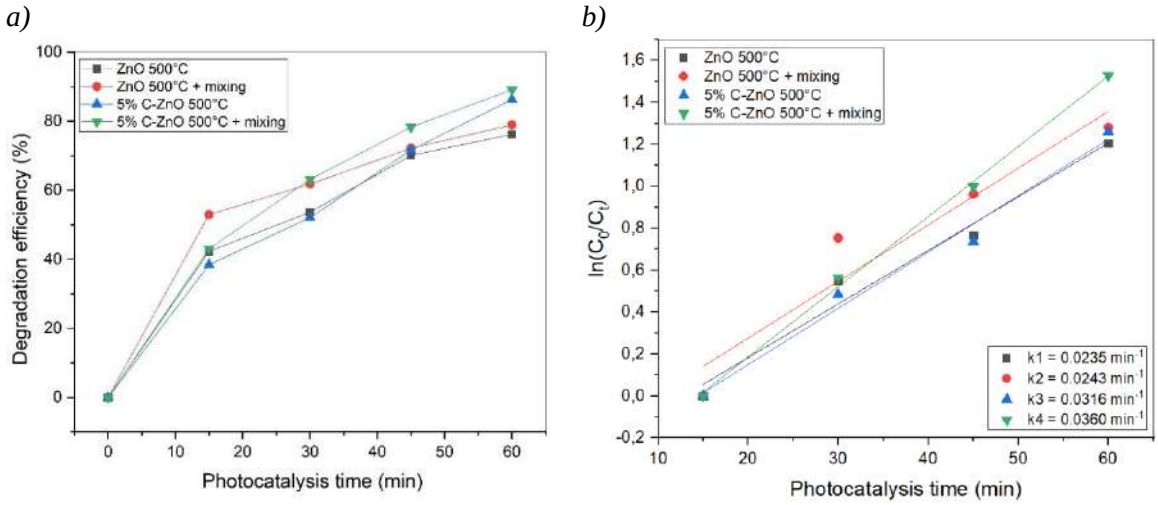

Figure 3 *a) Photocatalytic degradation efficiencies of synthesized nanoparticles; b) Kinetics curves of samples*

Overall, the enhanced performance of 5%C-ZnO can be attributed to the structural modifications and increased surface area resulting from carbon doping, which improves the efficiency and rate of photocatalytic degradation.

CONCLUSION

Facile and cost-effective mechanochemical synthesis of pure and C-doped ZnO nanoparticles was performed, and the prepared photocatalysts were used for photocatalytic degradation of methyl orange dye as a model pollutant. The results of the SEM and XRD studies were in agreement and showed that both samples were composed of nanoparticles. The XRD patterns illustrated that the C-ZnO-NP had a lower average crystallite size than the crystallite size of the pure ZnO-NPs, which indicates that the C-ZnO-NPs have a larger specific surface area as well as a larger number of defects, resulting in better overall photocatalytic properties. These alterations are more than just morphological; they confer enhanced photocatalytic efficiency to the C-ZnO-NP. Remarkably, a doping concentration of 5 wt% carbon enables the nanoparticles to achieve an 89.10% degradation rate of a 5 ppm methyl orange solution within the span of an hour, under neutral pH conditions. This efficiency not only underlines the potential for practical application in wastewater treatment but also makes the doped nanoparticles excellent candidates for environmental remediation.

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