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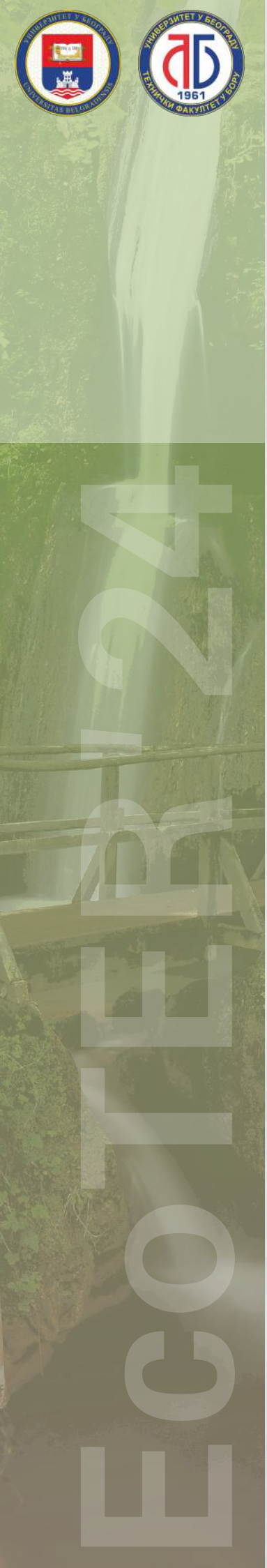

31st International conference

Ecological Truth & Environmental Research

Editor
Prof. Dr Snežana Šerbula

PROCEEDINGS

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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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INFLUENCE OF CALCINATION TEMPERATURE ON THE MORPHOLOGY, CHEMICAL COMPOSITION, AND STRUCTURE OF ZnO NANOPARTICLES

Sonja Stanković[1*], Vladan Nedelkovski[1], Dragos Buzdugan[2], Iosif Hulka[3], Milan Gorgievski[1], Snežana Milić[1], Milan Radovanović[1]

[1]University of Belgrade, Technical Faculty in Bor, V.J. 12, 19210 Bor, SERBIA

[2]Politehnica University of Timișoara, Faculty of Mechanics, M.V.S. 1, 300222 Timișoara, ROMANIA

[3]University Politehnica Timișoara, Research Institute for Renewable Energies, S.M.G. 138, 300501 Timișoara, ROMANIA

[*]*sstankovic@tfbor.bg.ac.rs*

Abstract

In this study, the influence of calcination temperature on the morphology, chemical composition, and structure of ZnO nanoparticles (ZnO–NP) was investigated. The ZnO nanoparticles were synthesized by a co–precipitation method. Zinc acetate dihydrate was used for the synthesis of zinc oxide nanoparticles. Thermogravimetric analysis (TGA) of the precursor was performed to determine the calcination temperature. The results of the TG analysis indicate high stability of ZnO at temperatures above 325°C. Therefore the prepared samples were calcined at temperatures of 400°C, 500°C, 600°C, or 700°C. The ZnO nanoparticles were morphologically and structurally characterized by X–ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersion spectroscopy (EDS). The results of the XRD analysis show that pure ZnO is obtained by the co–precipitation method and that the ZnO nanoparticles have a wurtzite structure. The average crystallite sizes of the materials calcined at 400°C, 500°C, 600°C and 700°C were 32.07 nm, 32.89 nm, 35.63 nm and 38.48 nm, respectively. As the calcination temperature increases, the crystallite size also increases. The results of SEM analysis show that the nano–sized particles were obtained by the co–precipitation method and that the calcination temperature significantly affects the size and morphology of the particles. The results of the EDS analysis show that pure ZnO was synthesized, which is consistent with the results of the XRD analysis.

Keywords: ZnO, nanoparticles, coprecipitation method.

INTRODUCTION

Urbanization and industrialization have contributed to the generation of large quantities of wastewater [1,2]. Eighty percent of wastewater is discharged into the environment without prior treatment. The discharge of wastewater into aquatic ecosystems without prior treatment is harmful to the environment and human health and is a growing problem worldwide [3–5]. For this reason, wastewater treatment is important for the protection of the environment and the preservation of human health.

There are a variety of traditional and non–conventional methods for wastewater treatment. However, the application of these methods is limited [6,7]. Photocatalysis is an efficient and

environmentally friendly process that can achieve complete mineralization of organic matter with low investment and operating costs, making it a promising method for wastewater treatment [5,8–10]. In recent years, researchers have focused on the synthesis of nano–sized photocatalysts for wastewater treatment. The nanomaterial that has attracted the most attention is zinc oxide, due to its stability, favorable photophysical properties, and antibacterial activity [7,11,12]. ZnO nanomaterials can be synthesized by various methods, including the sol–gel method, hydrothermal method, co–precipitation method, thermal decomposition method, and spray pyrolysis [7]. One of the simplest and most commonly used methods for the synthesis of ZnO nanoparticles is the co–precipitation method. In addition to the synthesis method, the size and morphology of ZnO nanoparticles, and thus their photocatalytic activity, are largely influenced by the calcination temperature.

In this paper, the influence of calcination temperature on the morphology, chemical composition, and crystallite size of ZnO nanoparticles synthesized by the co–precipitation method is investigated.

EXPERIMENTAL PART

Synthesis of ZnO nanoparticles

22 g of $Zn(CH_3COO)_2$ was dissolved in 100 ml of 80% ethanol. 0.05M NaOH was used to adjust the pH of the solution. The resulting solution (pH 12.86) was heated to 80°C and kept at a constant temperature until the clarified liquid separated from the precipitate. The suspension is filtered through filter paper (black tape). The resulting sediment is dried in a laboratory dryer at 80°C until it is dry. The dried sediment is transferred to a porcelain crucible and calcined at temperatures of 400°C, 500°C, 600°C or 700°C and labelled with the following markers: ZnO–400, ZnO–500, ZnO–600, or ZnO–700.

Characterization methods

The characterization of ZnO nanoparticles is essential to determine their structure and properties, as these can influence their photocatalytic activity in wastewater treatment. Various analytical techniques have been used to characterize zinc oxide (ZnO) nanoparticles, including thermogravimetric analysis (TGA), X–ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersion spectroscopy (EDS).

The thermogravimetric (TG) curve of zinc acetate dihydrate was recorded using the SDT Q600 simultaneous thermal analyzer from room temperature to 800°C at a heating rate of 10°C/min. X–ray diffraction (XRD) analysis was performed using an X'Pert³ powder diffractometer at a heating rate of 10°/min and an opening angle of 20–90°. SEM–EDS analyses were performed with the QUANTA FEG 250 SEM microscope.

RESULTS AND DISCUSSION

TGA

Thermogravimetric analysis was carried out to determine the calcination temperature. By analyzing the differential thermogravimetric curves shown in Figure 1, a two–stage mass loss was observed. In the first stage, in the temperature range from 56 to 104°C, about 16.53% of

the initial mass of the sample is lost due to the dehydration of the zinc acetate dihydrate. Similar results and observations were obtained by Ghule *et al.* [13], Horzum *et al.* [14], and Nguyen and Nguyen [15]. In the second phase in the temperature interval from 156°C to 325°C, a mass loss of 67.73% was observed due to the decomposition of zinc acetate to zinc oxide [14,15]. The thermogravimetric curves in the temperature range from 325 to 800°C revealed no weight loss, indicating complete decomposition of the precursor and the formation of highly stable ZnO nanoparticles [15].

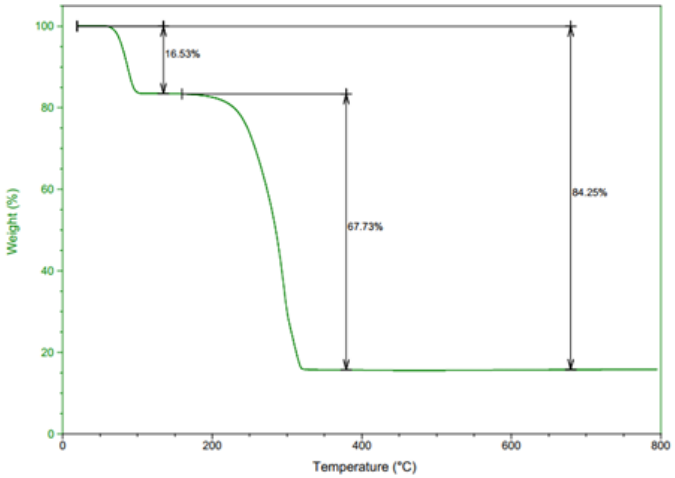

Figure 1 *Thermogravimetric analysis of $Zn(CH_3COO)_2{\cdot}2H_2O$*

XRD

The structure of the prepared ZnO samples was investigated using X–ray powder diffraction, and the diffractograms of the samples are shown in Figure 2. The diffraction peaks corresponding to (100), (002), (101), (102), (110), (103), (112), and (201) planes of ZnO are identical to those from the JCPDS database (JCPDS No. 036–1451) and indicate the characteristic wurtzite structure of ZnO [15–18]. Zincite was the only crystalline phase identified in all four samples analyzed. Not a single peak indicating the presence of impurities, Zn or $Zn(OH)_2$, was detected. This indicates that high–purity ZnO was synthesized by the coprecipitation method. Similar results and observations were obtained by Saravanan *et al.* [19], Baharudin *et al.* [20], Akpomie *et al.* [21], and Talam *et al.* [22]. The Debye–Scherrer equation (1) was used to determine the average cristallite size of ZnO nanoparticles:

$$D = \frac{k \cdot \lambda}{\beta \cdot cos\theta} \tag{1}$$

where: D – crystallite size (nm), k – constant (0.89), λ – wavelength of X–ray radiation (0.154 nm), β – full width at half maximum of the most intense diffraction peak (FWHM) (rad), θ – Bragg's angle (˚) [23]. The average crystallite size of the ZnO–400, ZnO–500, ZnO–600 and ZnO–700 samples was 32.07 nm, 32.89 nm, 35.63 nm and 38.48 nm, respectively. It can be concluded that the crystallite size of the ZnO–NP calcined at 400°C was the smallest and that the crystallite size increases with increasing calcination temperature.

This is probably due to the fusion of smaller crystallites into larger ones caused by high calcination temperatures [24].

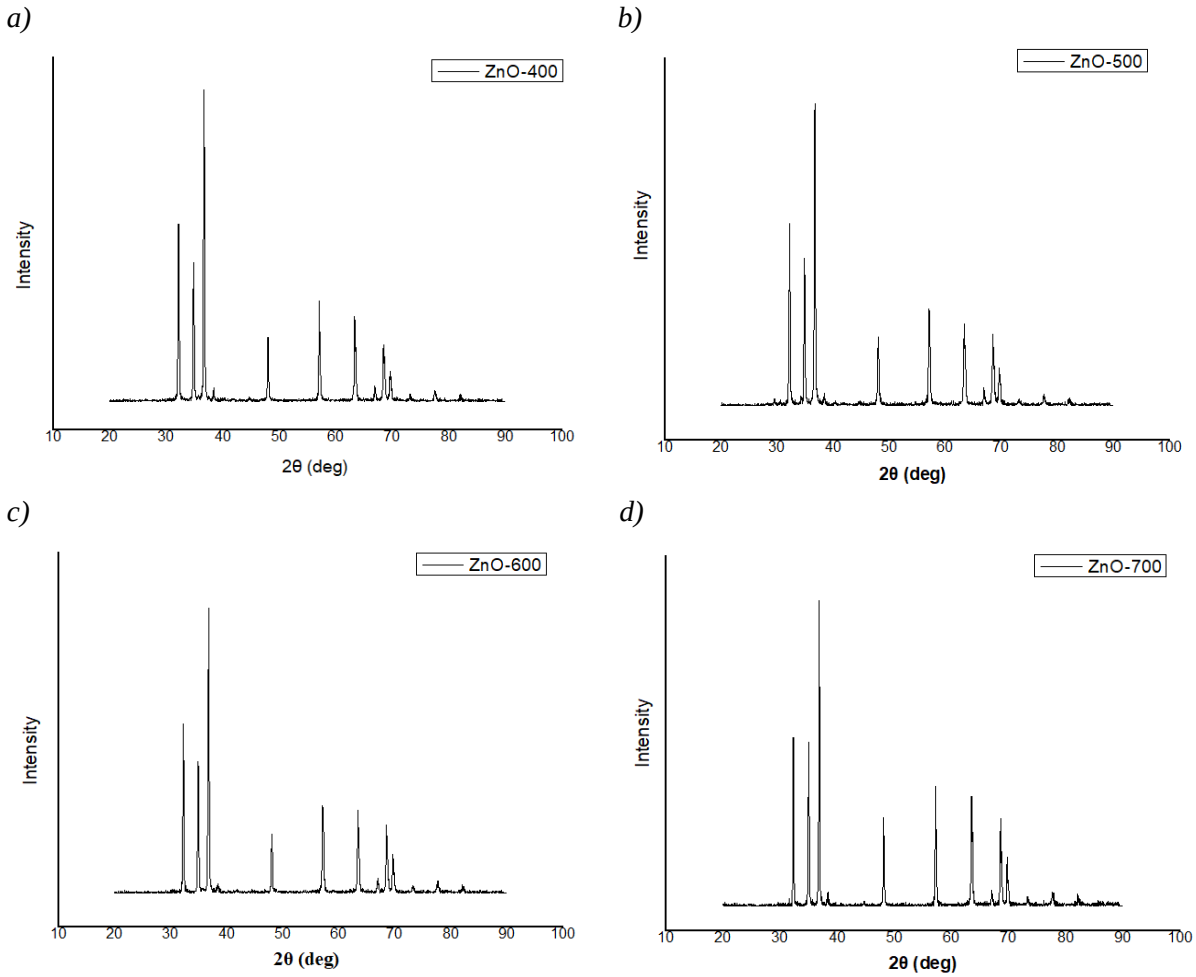

Figure 2 *Diffractograms of ZnO–NP calcined at: a) 400°C; b) 500°C; c) 600°C; and d) 700°C*

SEM–EDS

Scanning Electron Microscopy (SEM) was used to investigate the morphology and size of the ZnO nanoparticles. The results of the SEM analysis are shown in Figure 3 and indicate that the ZnO nanoparticles are heterogeneous in nature and agglomerate under certain conditions. The calcination temperature affects the morphology of the ZnO nanoparticles. Increasing the calcination temperature leads to a lower uniformity of the particles [20]. In samples calcined at temperatures of 400–600°C, spherical and nanorod–shaped particles dominate, while in the sample calcined at 700°C, hexagonal–shaped particles can be observed in addition to spherical and nanorod–shaped particles.

There is a correlation between the particle size and the crystallite size: as the calcination temperature increases, the size of the crystallites and particles increases [18]. In addition, as the calcination temperature increases, the pore volume increases, which can consequently have a positive effect on increasing the photocatalytic activity of the ZnO particles.

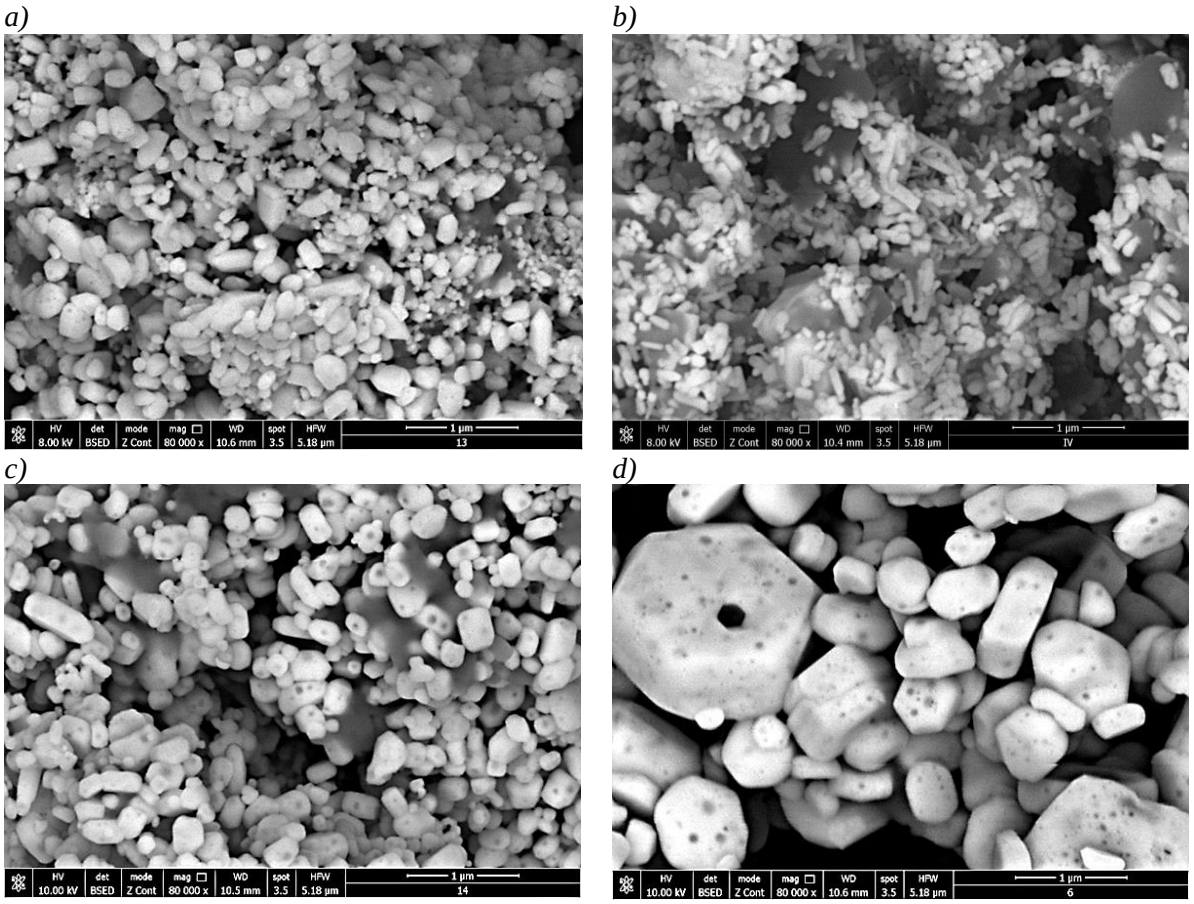

Figure 3 *SEM images of the ZnO nanoparticles thermally decomposed at a) 400°C; b) 500°C; c) 600°C; d) 700°C*

Energy dispersion spectroscopy (EDS) was used to determine the elemental composition. The results of the analysis (Figure 4) clearly show the strong peaks of the elements zinc (Zn) and oxygen (O). The absence of peaks of other elements indicates that pure ZnO was synthesized by the co–precipitation method, which is consistent with the results of XRD analysis. Similar results were obtained by Lad *et al.* [16], and Akpomie *et al.* [21]. The absence of impurities is very important for the further application of the synthesized ZnO NPs for wastewater treatment.

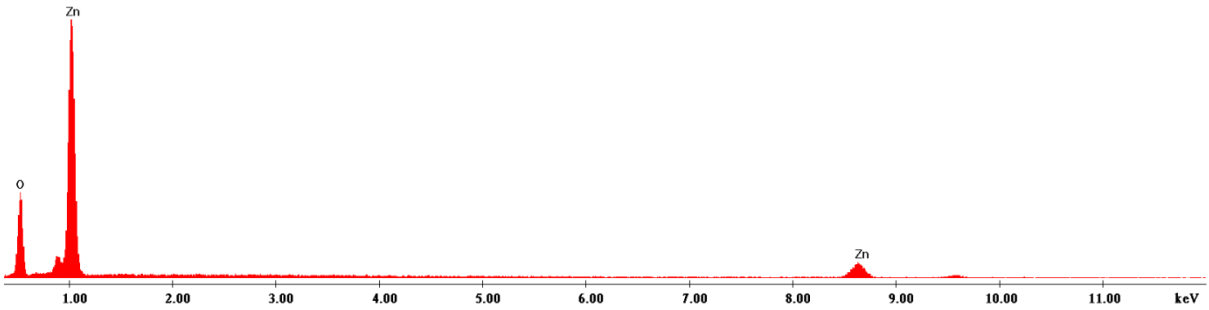

Figure 4 *Energy dispersive spectroscopy spectra of ZnO–NPs*

CONCLUSION

ZnO nanomaterials were successfully prepared by the co–precipitation method. The comprehensive characterization of the ZnO nanoparticles using SEM, EDS, and XRD techniques has provided valuable insights into the influence of calcination temperature in the range of 400–700°C on their structure, morphology, and chemical composition. The results show a wurtzite hexagonal structure of ZnO–400, ZnO–500, ZnO–600, and ZnO–700 with crystal sizes of 32.07 nm, 32.89 nm, 35.63 nm and 38.48 nm, respectively. The particles are heterogeneous in nature and the calcination temperature has a significant effect on the morphology of the particles. In samples calcined at temperatures of 400–600°C, spherical and nanorod–shaped particles dominate, while in the sample calcined at 700°C, hexagonal–shaped particles can be found in addition to spherical and nanorod–shaped particles. The particle size also increases with increasing calcination temperature. The results of the EDS analysis agree with the results of the XRD analysis and indicate that pure ZnO was synthesized by the coprecipitation method. From the presented results, it can be concluded that the calcination temperature significantly influences the morphology and size of the particles.

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