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Synthesis and Characterization of Zinc Oxide Nanostructures for Photocatalytic Applications

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Abstract

Zinc oxide has been widely investigated by many researchers due to their potential in an optoelectronic device. The problem statement focuses on the need for effective synthesis techniques using the co-precipitation method. This method aims to facilitate exact control of the size, shape, and morphology of ZnO nanostructures to improve and optimise their photocatalytic characteristics. The study aimed to synthesise ZnO nanoparticles by the co-precipitation technique and to examine the properties of ZnO nanoparticles under varying reaction concentrations and pH levels. Multiple methods exist for the synthesis of ZnO nanoparticles. This study focused on the co-precipitation synthesis process. The characterisation of ZnO nanoparticles was conducted using Field Emission Scanning Electron Microscopy (FESEM), X-ray Diffraction (XRD), and Ultraviolet-Visible Spectrophotometry (UV-Vis) to investigate the morphological characteristics of ZnO nanoparticles at varying reaction concentrations and pH levels. The optical and structural properties of the synthesized ZnO products have been evaluated. The nanorods that were grown were found to exhibit good optical properties. This method is environmentally friendly, cost-effective, and simple, making it a potential approach for large-scale production of devices and other applications.

1. Introduction

Synthesis and photocatalytic properties of ZnO nanostructures have garnered significant attention in the field of nanotechnology due to their unique characteristics and potential applications [1]. These nanostructures are synthesized through different techniques, including chemical vapor deposition, hydrothermal synthesis, hydrogen plasma-metal reaction, laser pyrolysis in the vapor phase, microemulsion, nonchemical microbial processes in the liquid phase, sol-gel methods, and ball milling in the solid phase[2]. Once synthesized, ZnO nanostructures exhibit excellent photocatalytic properties, making them highly desirable for environmental remediation, solar energy conversion, and wastewater treatment applications.

The ability of ZnO nanostructures to efficiently harness solar energy and facilitate chemical reactions through photocatalysis has sparked great interest among researchers and scientists. Furthermore, developing ZnO nanostructures with different morphologies and sizes has allowed for exploring their structure-function relationships, leading to a better understanding of their photocatalytic properties and potential applications. Additionally, the synthesis of ZnO nanostructures has been extensively studied to enhance their photocatalytic performance through doping, surface modification, and composite formation[3].

Zinc Oxide (ZnO) has a wide number of properties that make it an attractive material for various applications, particularly in the field of nanotechnology. The unique properties of ZnO, such as its wide bandgap, high optical transparency, and low toxicity, make it a promising candidate for developing nanomaterials with photocatalytic capabilities. The synthesis of ZnO nanostructures involves the manipulation of its morphology, size, and surface properties to tailor its photocatalytic performance. Materials classified as nanoscale are those whose minimum dimension is smaller than 100 nanometers. A nanometer is one-millionth of a millimeter or 100,000 times smaller than the diameter of a human hair. Nanomaterials are interesting because distinct optical, magnetic, electrical, and other properties occur at this scale. The field of electronics, medicine, environmental remediation, and other fields stand to benefit greatly from these emerging features [4]

In photocatalytic applications, due to their strong oxidizing power and stability, ZnO nanostructures play a crucial role in environmental purification, such as the degradation of organic pollutants and wastewater treatment [5]. However, traditional synthesis methods, such as vapor-phase synthesis and hydrothermal methods, present significant challenges, including high costs, complex procedures, and stringent temperature requirements [6]. These limitations hinder the large-scale production and practical application of ZnO nanostructures. The current methods for synthesizing ZnO nanostructures often result in high production costs, require sophisticated equipment, and involve complex, energy-intensive processes.

Although there are many previous reports, the major challenge in synthesizing ZnO nanostructures is achieving control over their size, shape, and morphology. This lack of control hinders the understanding and optimization of their photocatalytic properties, as these properties are highly dependent on the nanostructure characteristics. Thus, the main problem addressed in this project is the need for effective synthesis techniques of the co-precipitation method that can provide precise control over the size, shape, and morphology of ZnO nanostructures to optimize their photocatalytic properties. Additionally, comprehensive characterization techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV-Vis spectroscopy are essential to evaluate the structural, morphological, and optical properties of ZnO nanostructures. This study aims to develop and characterize ZnO nanostructures synthesized via the co-precipitation method to enhance their photocatalytic efficiency in environmental applications [6].

2. Experimental Details

2.1 Materials

Zinc oxide (ZnO) nanostructures were synthesized using a straightforward wet chemical method which are Zinc Nitrate Hexahydrate ($Zn(NO_3)_2{\cdot}6H_2O$) with a purity of at least 99% (HmbG Chemicals) at a concentration of 0.5 M and Sodium Hydroxide (NaOH) with a minimum purity of 98% (Sigma Aldrich) at a concentration of 1 M were prepared with deionized water.

2.2 Sample preparations

ZnO solutions were synthesized using the co-precipitation technique began with the preparation of 100 ml solutions of 0.5 M Zinc Nitrate Hexahydrate ($Zn(NO_3)_2.6H_2O$) and 1 M Sodium ZnO nanostructures were synthesized using the co-precipitation technique, beginning with the preparation of 100 ml solutions of 0.5 M Zinc Nitrate Hexahydrate (Zn(NO3)2·6H2O) and 1 M Sodium Hydroxide (NaOH) using deionized water. About 14.87 g of Zn(NO3)2·6H2O and 4 g of NaOH were dissolved in separate 100 ml volumetric flasks containing distilled water and stirred for 5 minutes at 600 rpm to ensure complete dissolution. The NaOH solution was then stirred vigorously at room temperature with a magnetic stirrer, and the zinc nitrate solution was gradually added drop by drop over 15 minutes while stirring continued for 2 hours. The resulting white precipitate was filtered, washed with deionized water, and dried to obtain ZnO powder, which was subsequently annealed at 200°C, 400°C, and 800°C to enhance its crystalline quality. Experiments were conducted to investigate the effects of pH value and concentration on the synthesis of ZnO nanorods. The synthesized ZnO nanorods were characterized using XRD for crystalline structural analysis, FESEM to study surface morphology and elemental composition, and UV-vis spectroscopy to examine their optical properties.

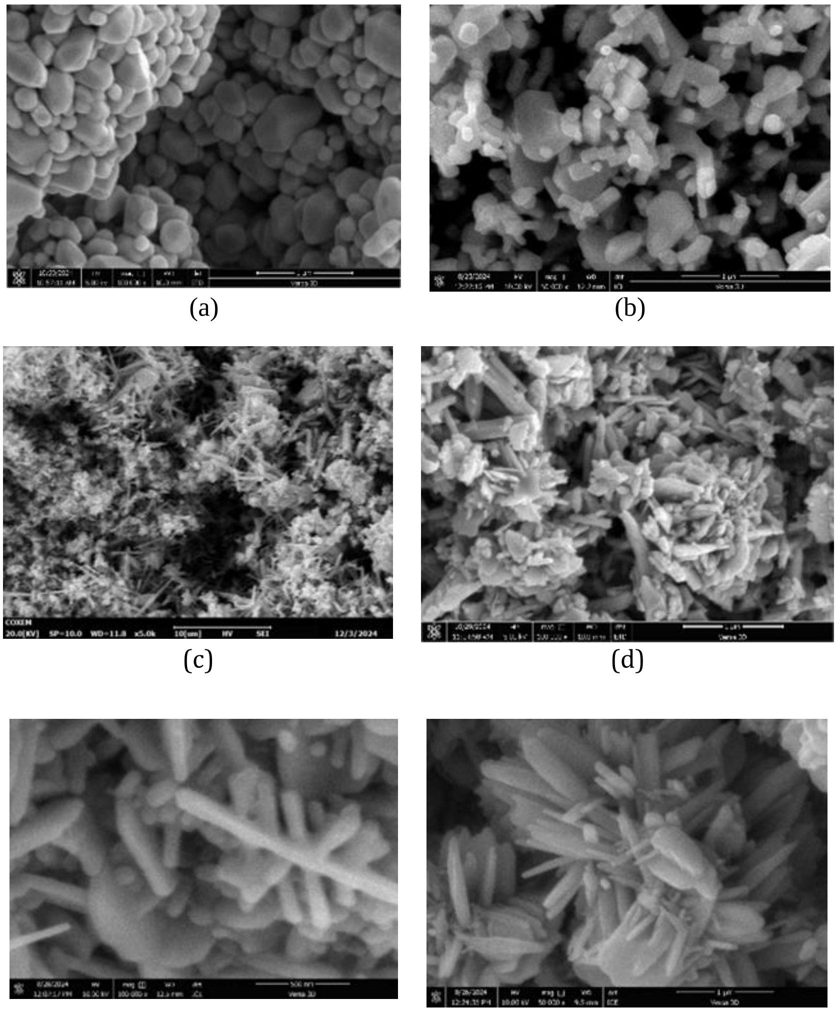
2.3 Sample characterizations

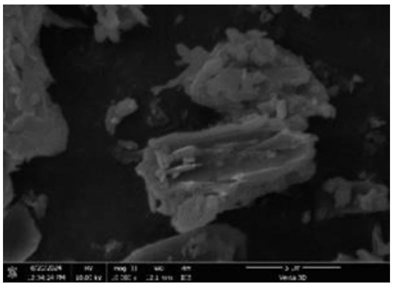
The biogenic zinc oxide (ZnO) nanoparticles were analysed using various analytical techniques to determine their optical, structural, and morphological properties. UV-Vis spectroscopy, X-ray diffraction (XRD), and field emission scanning electron microscopy (FESEM) were employed to characterize the synthesized nanostructures comprehensively. The morphological characteristics of ZnO nanoparticles were investigated using a Nova NanoSEM 450 (FEI) FESEM. Imaging was conducted at an accelerating voltage of 5–15 kV, with working distances of 5–10 mm. Magnifications ranged from 10,000× to 100,000× to observe size distribution, surface structure, and particle agglomeration. The crystalline structure was analysed using a Bruker D2 PHASER X-ray diffractometer. The XRD scan was performed in the 2θ range of 20°–80°, using a Cu-Kα radiation source (λ = 1.5406 Å) operating at 30 kV and 10 mA. The crystallite size was calculated using the Scherrer equation, revealing the degree of crystallinity and phase purity. The optical properties of ZnO nanoparticles were examined using a Perkin Elmer Lambda 950 UV-Vis Spectrophotometer. The absorption spectrum was recorded in the wavelength range of 100–800 nm. Baseline correction was performed, and the sample was dispersed in an appropriate solvent to ensure homogeneity.

3. Results and Discussions

3.1 Surface Morphology the Co-precipitate grown ZnO

Fig. 1 shows the FESEM image of ZnO nanoparticles synthesized by the co-precipitation method. According to [7] reported that the other size particles and shapes of ZnO nanoparticles with the particles ' hexagonal shapes and sizes of about 30- 57 nm. By comparing all the results of FESEM images of ZnO nanoparticles in Fig. 1, the affected size particles belong to the concentration of the precursor, which is zinc nitrate hexahydrate ($Zn(NO_3)_2.6H_2O$). Also, [8] mentioned that better surface morphology could be detected when the temperature increases and the concentration of zinc nitrate hexahydrate also increases.


(a) (b) (c) (d) (e) (f)



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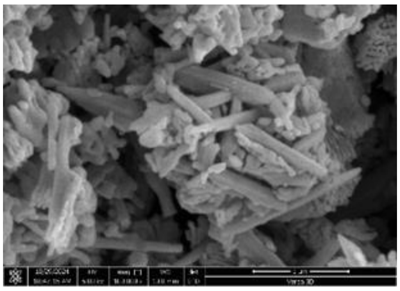

(h)

Fig. 1 FESEM images of ZnO nanostructures synthesized at different pH levels and concentrations: (a) pH 6 with 0.5 mM, (b) pH 6 with 1.0 mM, (c) pH 7 with 0.5 mM, (d) pH 7 with 1.0 mM, (e) pH 8 with 0.5 mM, (f) pH 8 with 1.0 mM, (g) pH 12 with 0.5 mM, and (h) pH 12 with 1.0 mM

Based on Fig. 1, numerous ZnO nanostructures with various combinations of pH and molar concentration may be created. The morphology and size of the ZnO nanostructure is controlled by the pH of the solution. This may be noticed when different pH levels of the solution produce various shapes of the ZnO nanostructure. For pH 6, the FESEM images indicate a significant presence of ZnO structures. At 0.5 mM, compact and aggregated formations dominate the surface, while 1 mM results in denser deposits with more well-defined crystalline features. These observations suggest that this pH level creates an ideal condition for ZnO nucleation and growth, leading to improved structural formation compared to lower pH environments [9].

In the case of pH 7, the images reveal further development of nanostructures as precipitation increases. The 0.5 mM concentration shows fibrous or porous morphologies, whereas 1 mM demonstrates clearer structural growth, potentially indicating the initial stages of nanorods or crystalline shapes. This pH provides a moderately supportive environment for the formation of ZnO structures. At pH 8, nanostructures are observed to be more refined and organized. Rod-like shapes become evident at 0.5 mM, while 1 mM leads to highly ordered structures,

with features resembling rods or needles dominating the morphology. These results indicate that pH 8 supports the robust development of ZnO nanostructures in various forms [10]. The pH 8 has been selected as the optimal condition because it enhances hydroxyl deprotonation, promotes ZnO nucleation, and facilitates nanorod growth. Studies indicate that alkaline conditions improve crystallinity and morphology, resulting in well-structured ZnO for photocatalysis. This ensures refined nanostructures with superior properties, making pH 8 ideal for high-performance applications according to [11].

When the pH reaches 12, the growth shifts toward more complex and advanced morphologies. Plate-like or layered structures emerge at 0.5 mM, suggesting a different growth mechanism compared to lower pH levels. At 1 mM, the nanostructures appear even more intricate, forming hierarchical or flower-like shapes. This demonstrates that higher pH conditions are particularly favourable for producing advanced ZnO structures, such as nano-flowers or fibers [12]. The FESEM images reveal nanostructures with widths ranging from 20 to 100 nanometres, emphasizing their nanoscale dimensions. These nanostructures display smooth and uniform surfaces, highlighting the effectiveness of the synthesis process. Nanoparticles exhibit a certain level of aggregation due to their elevated surface energy. This morphological characterization aligns with findings from research papers by [13], [14] which describe similar nanostructure properties in ZnO synthesized using sol-gel techniques.

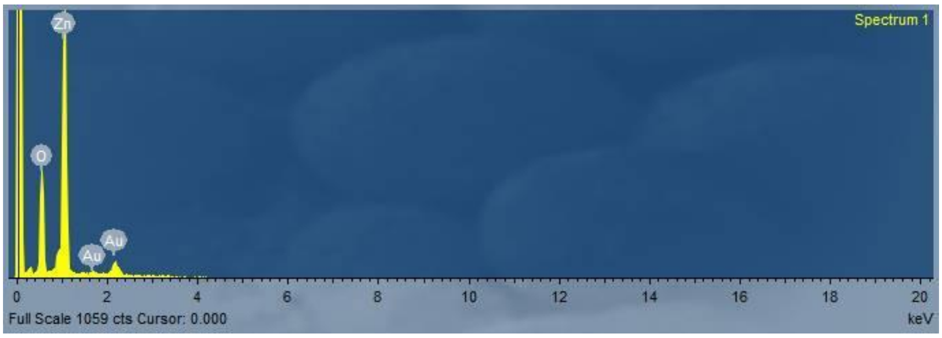

Fig. 2 The EDX analysis of Zn

Table 1 Data of ZnO obtained from EDX

Element	Weight %	Atomic %
Zn	19.43	49.12
O	8.00	46.92
Au	40.55	3.96
Total		100.00

Fig. 2 and Table 1 illustrate the sample's elemental composition as determined by EDX analysis. The table indicates that zinc (Zn) and oxygen (O) are the primary components, with atomic percentages of 49.12% and 46.92%, respectively, confirming the formation of ZnO in the sample. The presence of gold (Au) is also observed, with a weight percentage of 40.55%. This is attributed to the gold coating applied during sample preparation to improve conductivity for the EDX analysis. The high percentage of gold is not part of the ZnO compound but rather a result of this coating process. The data confirm that ZnO nanostructures were successfully synthesized, as indicated by the significant Zn and O concentrations.

3.2 X-ray Diffractometer (XRD) Analysis

X-ray Diffraction (XRD) is one of the machines to characterize the sample of ZnO nanoparticles. XRD is an important characterization device to determine the crystallographic structure and phases of materials [15]. From the X-ray Diffraction (XRD) analysis spectrum results of the sample ZnO nanostructures, as shown in Fig. 3, confirms the crystalline structure of the ZnO nanostructures. The XRD pattern exhibits sharp and well-defined peaks corresponding to the wurtzite hexagonal structure of ZnO, with characteristic peaks observed at 2θ values of approximately 2θ = 31.7°, 34.4°, 36.2°,47.5°, 56.6°, 62.8°, 69.9° which correspond to the (100), (002), (101), (102), (110), (103) and (112). As shown in Fig. 3, it is in the range of 30° to 80°, reflecting the nanostructured nature of the material. Moreover, the absence of impurity peaks, as illustrated in Fig. 3, indicates the high purity of the synthesized material. These results align with the findings of [16], [17].

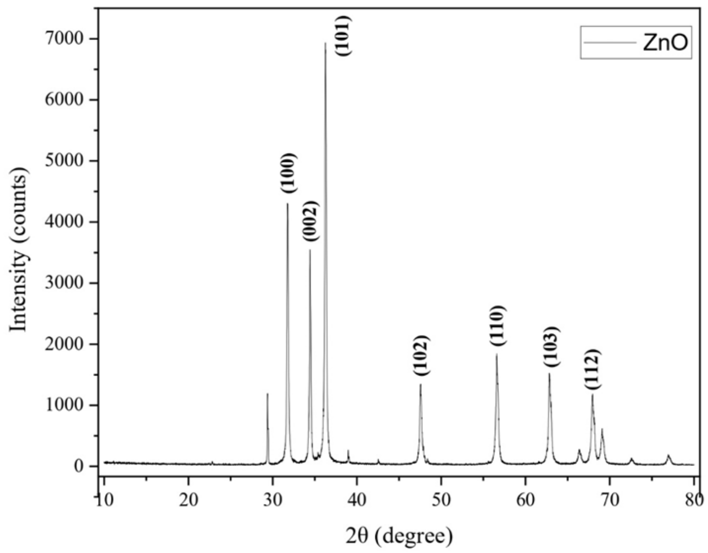

Fig. 3 X-ray diffraction pattern of ZnO nanoparticles

3.3 Absorption Properties by UV-Visible Spectrometer

The UV-Vis absorption spectrum of the synthesized ZnO nanorods, as shown in Fig. 4, exhibits an absorption peak at approximately 310 nm with a maximum absorbance of 3.355eV. This peak is attributed to the intrinsic band-edge absorption of ZnO, signifying its characteristic optical behaviour. The observed blue shift in the absorption edge compared to bulk ZnO (typically in the range of 360–380 nm) is a clear indicator of quantum confinement effects, which arise due to the reduced size of the nanorods at the nanoscale. This size-induced shift enhances the optical band gap energy and indicates superior optical properties [18].

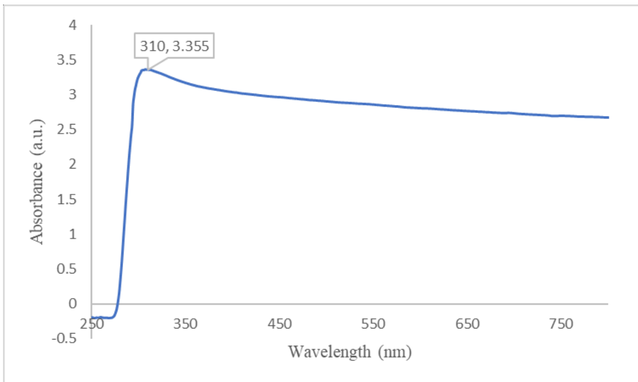

Figure 4 UV–vis absorption spectra of ZnO nanostructures

According to [19], ZnO nanostructures commonly display strong UV absorption with a band-edge transition, and their optical band gap can be calculated using methods such as the Tauc plot, yielding values around 3.37 eV. The blue-shifted absorption edge observed here aligns with this trend, confirming the quantum confinement effect in these nanorods. The absence of additional peaks in the spectrum indicates high sample purity, further demonstrating the excellent optical quality of the ZnO nanorods. Such properties make these nanostructures highly suitable for applications in UV photodetectors, light-emitting diodes, and other optoelectronic devices [20]

4. Conclusion

This review shows that the co-precipitation method synthesized ZnO nanoparticles (ZnO-NPs). Previous research and literature show that several synthesis approaches, including sol-gel procedures, hydrothermal methods, and chemical vapor deposition, have demonstrated efficacy in producing ZnO nanostructures with sizes, morphology, and crystallinity. The primary focus is to characterize the ZnO nanoparticles in each sample with varying concentrations and pH levels. This is validated through XRD analysis, which confirms the successful synthesis of ZnO particles using the co-precipitation method. Additionally, FESEM characterization was conducted to analyze the surface morphology of the samples. The results revealed that ZnO nanoparticles synthesized at pH 8 exhibited the most uniform morphology, with well-defined nanostructures, indicating that this pH level is optimal for producing high-quality ZnO particles. Furthermore, UV-vis analysis showed the highest absorbance for ZnO nanoparticles at a wavelength of approximately 310 nm. These findings confirm that synthesizing ZnO nanoparticles using the co-precipitation method produces high-purity ZnO with desirable nanostructural features, making them suitable for applications in photocatalysis, optoelectronics, and sensing technologies.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design, data collection, analysis and interpretation of results:*** *Nur Nabilah Mansor, Amira Saryati Ameruddin. All authors reviewed the results and approved the final version of the manuscript.*

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