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Original Research Article

Synthesis, Structural Characterization and Dielectric Study of Zinc Oxide Nanoparticles Prepared Via Co-Precipitation Method

Muhammad Abdul Salam[1]*, Rida Tariq[2], Muhammad Saad Aslam Qazi[3], Muhammad Haseeb[4], Fouzia Hameed[5], Muhammad Umer Farooq[6]

[1]Department of Chemistry, Khushal Khan Khattak University, Karak, Pakistan
[2]Department of Chemistry, COMSATS University Islamabad, Islamabad 44028, Pakistan
[3]Ruhr University Bochum, Germany
[4]Department of Physics, University of Agriculture, Faisalabad 38000, Punjab, Pakistan
[5]Department of Chemistry, The University of Lahore, Pakistan
[6]Department of Chemistry, University of Engineering and Technology (UET), Lahore 54590, Pakistan

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***Corresponding author:** Muhammad Abdul Salam
Department of Chemistry, Khushal Khan Khattak University, Karak, Pakistan

Abstract

In this study, zinc oxide nanoparticles were produced using a special method (co-precipitation), which had a hexagonal shape, in which zinc acetate serving as a starting material. The structure and optical characteristic were examined using technique X-ray diffraction (XRD), IR spectroscopy, and UV-Visible absorption spectroscopy. The XRD analysis confirm the formation of zinc oxide (ZnO) particles and via Scherrer equation lattice parameters and average crystallite size was find out. Furthermore, the dielectric properties like permittivity and dielectric loss of ZnO were analyzed by different frequencies and temperature to understand the potential of electronic application of Zinc Oxide.

Keywords: Zinc oxide nanoparticles, Co-precipitation method, XRD, UV-Vis spectroscopy, IR spectroscopy, Crystallite size, Dielectric properties.

1. INTRODUCTION

Zinc oxide, because it is non-toxic and assist in the flow of electricity, is regarded very important in research today. It has several special properties, such as a large band gap of about 3.37 eV, good electrical conductivity, strong energy particles of 60 meV, fast electron movement, and high resistance to heat and pressure. These characteristics make ZnO highly adaptable for numerous applications. Moreover, zinc oxide nanoparticles exhibit excellent optical transparency, making them suitable for use in electronic devices, UV lasers, solar cells, photocatalytic system, gas sensors, and field emission technique [1–4].

Beyond their optoelectronic advantage, zinc oxide shows great potential toward environmental remediation because of its eco-friendly nature. It has strong antibacterial properties and very good photocatalytic activity as well [5,6]. The original physicochemical properties of ZnO can be controlled properly via doping, like adding transition metals, or by surface modification, like coating with oxides. These changes can affect the material's energy levels, making it easier for electrons to move from the lower to the higher energy band. This is important for improving how well the material works in light-based reactions and electronic devices [7,8].

Numerous synthesis strategies have been investigated by researcher. For instance, Karam *et al.,* prepared TiO_2-coated ZnO nanoparticles and found that when the TiO_2 layer is thicker or the number of layers is increased, the surface area also increases, especially after reaching 1 micron in size [9]. Kumari *et al.,* made nitrogen-doped nanoparticles using the chemical precipitation method. They stated that nitrogen doping changes the crystalline size and optical properties. XRD confirmed that the structure was impurity-free wurtzite [10]. Rochman *et al.,* utilize the sol-gel method and noted that increasing the pH level led to greater particle

agglomeration [11]. Detta *et al.,* synthesized ZnO nanoparticles using green synthesis with *Parthenium hysterophorus* leaf extract. The particles were in spherical and cylindrical shapes, with sizes between 16–45 nm [12]. Sutradhar *et al.,* made ZnO nanoparticles using tomato extract through thermal and microwave methods. They said this method is eco-friendly and very suitable for photovoltaic applications [13].

Mohan *et al.,* compared ZnO nanoparticles made by the conventional method and the surfactant-assisted method. The particles synthesized with PVA surfactant were in the nanometer range, while the conventional method produced micron-sized particles [14]. McLaren *et al.,* studied the photocatalytic behavior of different crystal surfaces of ZnO and reported that the fabrication process affects particle shape and size [15]. Ahmed *et al.,* prepared Mn-doped ZnO particles and observed that Mn doping improves the lattice parameters and magnetic properties [16]. Theweesaeing *et al.,* synthesized copper-doped zinc oxide nanoparticles via co-precipitation and confirmed a hexagonal structure using XRD analysis [17].

Recently, nanoscale metal oxide nanostructures have drawn significant interest due to their unique electrical, magnetic, optical, and chemical behavior, which differ from their bulk form [18]. Especially, nanoscale ZnO offers specific advantages such as higher surface area, high porosity, low toxicity, and low-cost synthesis [19]. Because of all these reasons, it has become a very good candidate for catalysis [20], photocatalysis, UV-protective films, and chemical sensors [21–24].

For the preparation of ZnO, different synthetic methods were used such as thermal decomposition, vapor phase deposition, sol-gel, spray pyrolysis, and precipitation techniques, so that the morphology and size can be uniform [25–27]. Among all these methods, the precipitation method is considered suitable because of its simplicity, low cost, and applicability for large-scale production. This method does not require any complex apparatus or expensive precursors [28].

Despite extensive research, their remain a knowledge gap, regarding how variation and co-precipitation parameters influence the structural, morphological, and optical properties of ZnO, particularly when the work is done in a simple and low-cost laboratory. This is a very important gap because the co-precipitation method is recognized as a very easy, low-cost method and is used on a large scale, making it ideal for real-world applications.

Our research addresses these issues and contributes not only to academic knowledge but also to advancements in technology. In this research, we have shown how the properties of zinc oxide nanoparticles can be controlled using a low-cost method. This is necessary to increase the effective use of ZnO In areas like breaking down pollutants with light and cleaning up the environment, UV protection, and gas sensing. This study on zinc oxide not only contributes new information to current studies on zinc oxide nanoparticles but also provide guidance for understanding and advancing future green and sustainable nanotechnology. It is important to address this gap, as it show a strong connection between research done in lab and industrial applications, a connection is usually discussed but rarely actually use.

In this study, Zinc oxide nanoparticles were synthesized using co-precipitation technique, X-ray diffraction (XRD), infrared (IR) spectroscopy, and ultraviolet-visible (UV-Vis) spectroscopy were used to examine the resulting nanostructures to determine their structure and optical properties.

2. MATERIALS AND METHODS

I. Materials:

Zinc acetate dihydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$) and sodium hydroxide (NaOH) were used as precursors to synthesizes zinc oxide nanoparticles. These reagents were taken from the college laboratory and utilized as received, without any further purification. In every step of the synthesis and washing process the deionized water was utilized.

II. Synthesis of ZnO nanoparticles:

To prepare a 0.5 M zinc acetate solution, 10 grams of zinc acetate di hydrated $(CH_3COO)_2 \cdot 2H_2O$ were dissolved in 50 mL of deionized water. The solution was stirred at room temperature (approximately 25 ℃) for about one hour to ensure it complete dissolution. In a separate preparation, 4 grams of sodium hydroxide (NaOH) were dissolved in 50 mL of deionized water to obtain a 2 M NaOH solution, maintaining a pH close to 14.

This basic solution was then gradually added to the zinc acetate solution under constant stirring. The resulting mixture was stirred for an additional two hours at room temperature, during which a white gel-like precipitate formed. The mixture was left to stand at room temperature for 24 hours to allow the precipitate to fully settle.

The clear supernatant was gently decanted, leaving behind the precipitate, thoroughly rinsed multiple times with deionized water until a neutral pH was achieved. Once washing was complete the precipitate was dried in a laboratory for two hours. The resulting dried material was then finely ground using an agate mortar. To obtain pure zinc oxide nanoparticles the powder was subsequently calcined in a muffle at 500 ℃ for 2 hours in a muffle furnace.

3. CHARACTERIZATION

Following sample preparation, various analytical technique was employed to investigate its

structural and optical properties. These techniques included XRD, FTIR, UV-Vis, and TEM. A short explanation of each technique is given below.

a. X-Ray Diffraction (XRD):

X-rays are form of electromagnetic radiation with wavelength near 1 angstrom (10^{-10} meter). They fall between gamma rays and ultraviolet rays in the electromagnetic spectrum, and their wavelength is on the order of atomic dimensions, making X-rays especially useful for examine the internal atomic structure of crystalline materials. Since their discovery in 1895, X-rays have enabled scientist to analyze how atoms are arranged within crystals.

When X-rays interact with a crystalline substance, they produce a characteristic diffraction pattern arises when difference in the path length of the reflected X-ray correspond to an integer multiple of the X-ray wavelength. This condition is described as Bragg's Law, which is mathematically expressed as:

$n\lambda = 2d \sin(\theta)$

Where:

- n is a positive integer (e.g., 1, 2, 3, …)
- λ represent the wavelength of the incident rays (commonly 1.54 Å for copper)
- d is the distance between adjacent atomic planes in the crystal,
- θ is the angle of the incident at which the X-ray beam strikes the atomic planes.

By changing the angle (θ), we can find different d-values in the sample. This angle and the intensity of the diffracted rays are recorded using the XRD machine, resulting in a unique pattern similar to a fingerprint, which is characteristic of each material.

Almost 95% of solid materials are crystalline, and each crystal has its own specific XRD pattern. Today, more than 50,000 inorganic and organic crystal patterns have been recorded and are used as reference data. XRD is not only implies for the material identification, but also study to its structure, like how atoms are arranged, the distance between them, and the bond angles. That's why XRD is very important in material science and solid-state chemistry.

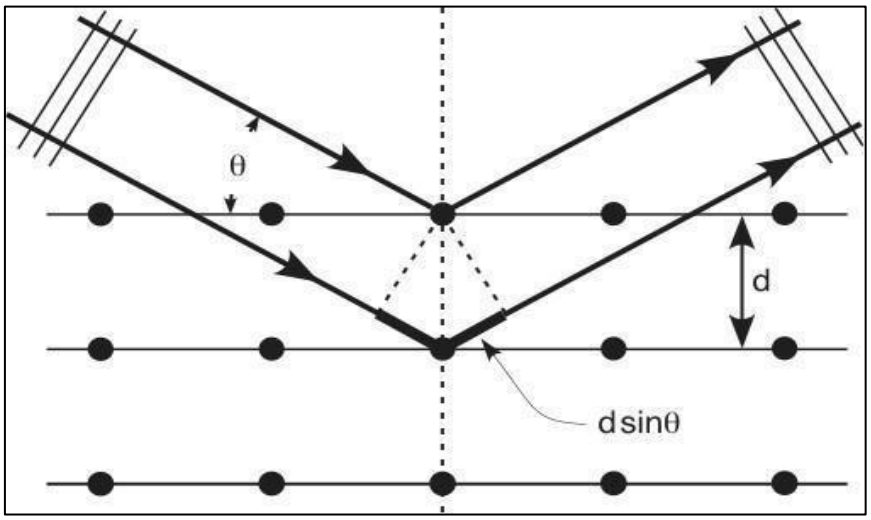

Figure 1.1: X-rays reflection from two planes of atoms in a solid.

b. X-ray Diffraction (XRD) Analysis:

The structural properties of the synthesized zinc oxide nanoparticles were analyzes through X-ray Diffraction (XRD). The obtained diffraction confirmed that all the observed peaks correspond to the hexagonal wurtzite phase of ZnO, indicating the successful formation of the desired crystal structure. This pattern was compared with the Penny Mathumba *et al., Mater. Res. Express* **11** 075011 [29], where every observed phase exactly matched the crystalline phases. No extra peaks of any impurity phases such as Zn $(OH)_2$, ZnS, or $ZnCO_3$ were found, which confirm that the prepared sample has no unwanted secondary phases.

The XRD results also shows that the nanoparticles have a well-crystallized, without any major lattice distortion or amorphous materials. These properties of zinc oxide are important for uses where structural stability, purity, and good crystal order directly affect functional performance, such as in photocatalysis, UV absorption, and gas sensing. The sharpness and intensity of the diffraction peaks, along with the narrow full width at half maximum (FWHM), indicate that the synthesized particles are within a nanometer scale. The average crystalline size can be estimated using the Debye–Scherrer formula. These results suggest that the zinc oxide nanoparticles produced through the co-precipitation method have a uniform crystal structure, high purity, and narrow size distribution.

c. UV-Visible Studies:

The optical properties of the synthesized ZnO nanoparticles were examined using UV-Vis spectrophotometer (e.g., Shimadzu UV-1800) in the wavelength range of 200–800 nm at room temperature (Figure 1.3). The spectrum displayed a pronounced absorption edge around 220 nm, which is the characteristic of zinc oxide nanoparticles. The sharp and high intensity of this absorption features suggest the formation of nanosized particles. This behavior can be attributed to the wide direct band gap of ZnO and the influence of quantum confinement, which enhance their optical response [30].

d. FTIR Spectrum:

FTIR spectroscopy was employed to identify the functional groups and bonding characteristics synthesized ZnO nanoparticles. The recorded spectrum (Figure 1.2) exhibited a clear band within the region of 800–1300 cm^{-1}, which is attributed to Zn-O stretching vibration. The presence of this characteristic peak verifies the successful formation of ZnO nanoparticles and confirms metal-oxygen bonding in the material [31].

4. RESULTS AND DISCUSSION

a. FTIR Spectra:

The FTIR spectrum, shown in Figure 1.2, indicates the presence of bonds between zinc and oxygen. A broad absorption peak was observed around 457 cm^{-1}, along with a shoulder peak near 545 cm^{-1}; both are characteristic of Zn–O stretching vibrations and confirm the Zn–O bond in zinc oxide nanoparticles [31]. The rest of the spectrum was smooth, but a minor small peak was observed due to the presence of atmospheric carbon dioxide gas, which may have been adsorbed during sample preparation or exposure to air.

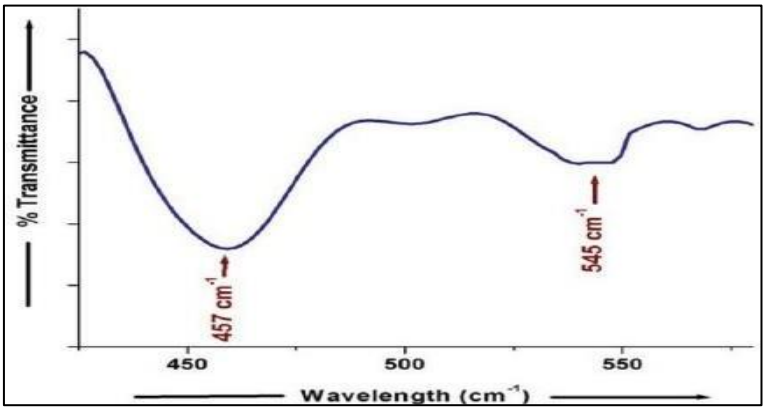

Figure 1.2: FT-IR spectrum of the zinc oxide nanoparticles

b. UV-Visible Spectroscopy:

The optical behavior of the synthesized zinc oxide nanoparticles was examined using UV-visible absorption spectroscopy, and the result are present in Figure 1.3. The absorption spectrum displays a strong and sharp absorption band at 355 nm, indicating the band-edge absorption of zinc oxide nanoparticles [32]. Additionally, a distinct excitonic peak is observed at 258 nm, assigned to the quantum size effect, as it appears at a shorter wavelength than the bulk ZnO band gap of 358 nm (Eg = 3.46 eV). These sharp and distinct peaks show that the synthesized nanoparticles are monodispersed, meaning they have a uniform size distribution [33].

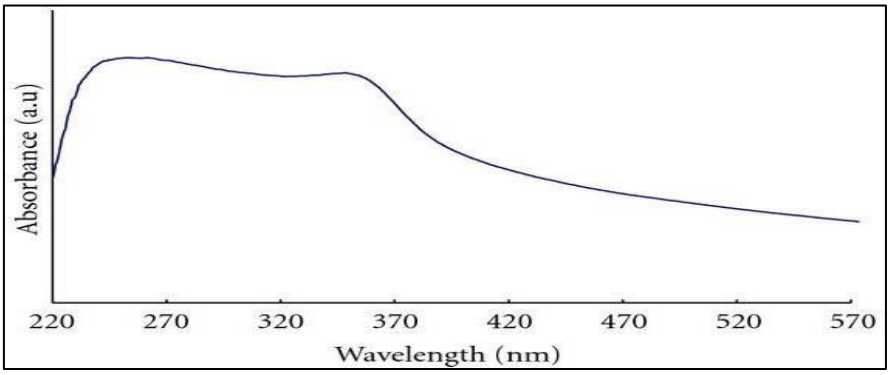

Figure 1.3: UV-Vis spectrum of the zinc oxide nanoparticles

c. XRD Analysis:

The crystal structural and phase purity of the synthesized zinc oxide nanoparticles were examined through X-ray diffraction (XRD), as illustrated in Figure 1.4. The diffraction exhibits well-define peaks corresponding to the (100), (002), (102), (110), (103),

(020), (112), and (201) planes, which are indicative of the hexagonal wurtzite crystal structure of zinc oxide nanoparticles [34]. Among these, the (101) shows the highest intensity, reflecting a high degree of crystallinity.

The average size was estimate using the Debye–Scherrer equation and was determined to be approximately 22.29 nm. Additionally, the calculated lattice constant was a = b = 3.2430 Å and c = 5.1950 Å, which align well with previously reported standard values [35], confirming the formation of a single-phase, impurity-free ZnO nanoparticles hexagonal symmetry.

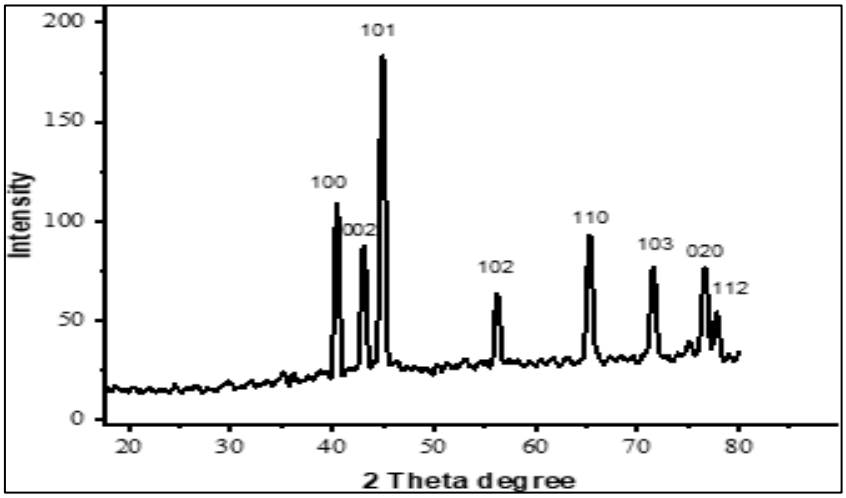

Figure 1.4: XRD pattern of ZnO nanoparticles synthesized in this study

5. CONCLUSION

Zinc oxide (ZnO) nanoparticles were successfully synthesized with a hexagonal wurtzite structure using the co-precipitation method. XRD analysis confirmed the crystalline wurtzite phase of zinc oxide nanoparticles, with the average particle size calculated using Scherrer equation as 15.87 nm (at 100 ℃) and 18.82 nm (at 200 ℃) [36]. From the UV-Visible spectroscopy, a sharp absorption peak was observed above 200 nm for zinc oxide nanoparticles, representing their nanoscale size and optical properties. The FTIR spectrum shows peaks in the range of 600–400 cm^{-1}, corresponding to Zn–O bonding. Dielectric studies show that by increasing frequency, both the dielectric constant and dielectric loss decrease, while the conductivity increases. This behavior supports the semiconducting nature of ZnO.

Overall, these finding demonstrate the synthesized zinc oxide nanoparticles possess excellent optical and electrical properties, which can be beneficial in the future for optoelectronic devices and nanotechnology applications.

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