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Synthesis and Characterization of ZnO Nanoparticles via Co-Precipitation Method

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ABSTRACT

Zinc oxide (ZnO) nanoparticles were prepared with an easy and relatively inexpensive co-precipitation route using zinc acetate dihydrate as the zinc source and sodium hydroxide (NaOH) as the precipitating agent. X-ray diffraction (XRD), fourier-transform infrared spectroscopy (FT-IR), and ultraviolet-violet (UV-Vis) spectroscopy were used to systematically characterize the resulting nanoparticles by studying their structural and optical properties. Based on the XRD, it was determined that a pure crystal structure of hexagonal wurtzite was obtained and that no secondary crystals, i.e. very high purity of the phase, were present. Debye Scherrer equation was used to determine the average crystallite size, which was found to be at nanoscale range confirming the successful formation of nanocrystalline ZnO. The distinct and strong absorption and excitonic peaks at around 355 nm and around 258 nm respectively, observed in the UV- Vis absorption spectrum, reveal the mono-dispersity and the size of nanoparticles. The FT-IR also confirmed the presence of the ZnO nanoparticles but showed strong absorption bands at range of 400-600 cm-1 which could be attributed to the Zn-O vibrations. Further, dielectric properties of the prepared ZnO nanoparticles were studied with respect to frequency and temperature. The results indicated that dielectric constant and dielectric loss decreased with frequency similar to the finding in

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semiconducting oxide materials.It has been depicted in the paper that co-precipitation technique is a good method to obtain ZnO nanoparticles with a desired structure and optical properties. The findings have shown ZnO nanomaterials could be utilized in other applications as optoelectronics, sensors and dielectric devices due to its stability, diverse band gap and prospective dielectric response.

Keywords: Zinc Oxide Nanoparticles, Co-Precipitation Method, XRD, FT-IR, UV-Vis Spectroscopy, Dielectric Properties, Wurtzite Structure, Nanocrystals, Optical Absorption, Scherrer Equation

Introduction

Zinc oxide (ZnO) is among the top II-VI semiconductor materials, which have received much research attention due to its excellent physical and chemical properties. These include large direct band gap of approximately 3.37 eV, large exciton binding energy of 60 meV, high thermal and mechanical stability and high optical transparency. These features make ZnO a very viable material in a wide variety of applications, especially optoelectronic devices, gas and chemical sensors, photocatalysis, solar energy collection systems, and environmental remediation systems.

The nanostructured variant of ZnO has shown better performance in the last couple of years, in large part because of its higher surface-to-volume ratio, quantum confinement phenomena, and because of greater control of the electronic and optical properties of the material. ZnO nanoparticles exhibit superior reactivity, light absorption and charge carrier dynamics unattainable in prior nanotechnology and materials science applications.

A number of synthetic techniques have now been developed to make ZnO nanoparticles that comprise sol-gel processing, hydrothermal synthesis, thermal decomposition and vapor-phase deposition. Co-precipitation technique is one such highly successful and environmentally innocuous path amongst them. The advantages of the technique include that they are easy to operate, economical, scalable, and do not require use of complex instrumentation and other hazardous reagents. Moreover, it enables the simplified regulation of the diameter of the particles and shape, which is of utmost significance in terms of matching the characteristics of the materials to the specific use.

In this paper, ZnO nanoparticles have been prepared using co-precipitation method and their characteristics have been established to quantify structural, optical and dielectric characteristics. It shows that good quality ZnO nanomaterials synthesized in this way can be utilized in electronics, optical and catalytic applications.

Literature Review

Due to their distinctive structural, optical and electronic characteristics, zinc oxide (ZnO) nanoparticles have been extensively studied concerning their production, modification and applications. There are many methods of synthesis and doping to alter the properties of ZnO to suit particular functionality needs.

In this study, Karam et al. (2018) examined TiO2 coated ZnO nanocrystals and discovered that surface area of the composite and uniformity, one of the determining factors in the operation of the dye sensitized solar cell, increased with the thickness of

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the TiO2 layers. Synthesizing nitrogen-doped ZnO nanoparticles, Kumari et al. (2015) applied the chemical precipitation method and noted that crystallite size grew with the addition of nitrogen. According to the paper, doping was used to grow a phase of wurtzite and alter its optical characteristics. Green synthesis pathways have been of interest because they are more environmentally friendly and sustainable. A study by Sutradhar and Saha (2016) demonstrated the synthesis of ZnO nanoparticles with tomato extract under microwave irradiation and its possible application in photovoltaic. Likewise, Parthenium hysterophorus leaf extract was used to prepare ZnO nanoparticles by Datta et al. (2017) and exhibited antimicrobial activity of great significance.

Rochman and Akwalia (2017) optimized the sol-gel technique by varying pH and found that increased pH values resulted in decreased agglomeration and homogenous formation of nanoparticles. Atias et al. (2010) also observed the same when they showed increased crystallinity and refined particles under alkaline pH conditions.

It has been repeatedly reported by other researchers that alternative synthesis routes lead to the formation of a hexagonal wurtzite structure, which has been verified by the X-ray method (XRD) (Thaweesaeing et al., 2013; Khalil et al., 2014). By showing that shape and size have a strong impact on photocatalytic activity of ZnO nanocrystals, McLaren et al. (2009) have proven the relevance of controlled synthesis.

Additionally, a recent study by Ahmed (2017) on ZnO doped with Mn and by Hameed et al. (2016) on ZnO doped with Nd has demonstrated that it is possible to tune the magnetic, optical, and structural properties successfully through doping. All that adds up to the fact that the ZnO nanostructures are versatile and that the conditions under which they grow can have a crucial impact on the end product.

Overall, we can say based on the literature that co-precipitation technique is a scalable, low-cost and efficient technique to make ZnO nanoparticles with tunable properties and can be used in other applications.

Materials and Methods

Materials

Zinc acetate dihydrate (Zn(CH3COO)2·2H2O)(Zn(CH₃COO)₂·2H₂O)(Zn(CH3COO)2·2H2O) and sodium hydroxide (NaOH) were used as precursors for the synthesis of zinc oxide nanoparticles. All reagents were of analytical grade and obtained from existing laboratory stock. Deionized water was used throughout the experiment for solution preparation and washing procedures.

Synthesis of ZnO Nanoparticles

Zinc oxide (ZnO) nanoparticles were synthesized via the co-precipitation method using the following procedure:

Preparation of Zinc Precursor Solution:

A total of 10 g of zinc acetate dihydrate was dissolved in 50 mL of deionized water. The solution was continuously stirred using a magnetic stirrer for 1 hour at room temperature (~25 °C) to ensure complete dissolution.

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Preparation of Alkaline Precipitating Agent:
In a separate beaker, 4 g of sodium hydroxide was dissolved in 50 mL of deionized water to prepare a basic solution. The solution was stirred until fully homogenized.
Precipitation Reaction:
The NaOH solution was added dropwise to the zinc acetate solution under constant stirring, maintaining the pH at approximately 14. Stirring was continued for 2 hours, during which a white gel-like precipitate gradually formed.

Aging and Washing:
The reaction mixture was allowed to stand undisturbed overnight at room temperature to complete the aging process. The resulting precipitate was separated and washed multiple times with deionized water until a neutral pH was achieved to remove any residual ions or unreacted chemicals.

Drying and Calcination:
The dried product was put in the hot air oven at 120 °C and dried in 2 hours. The powder thus obtained was dried in an agate mortar, followed by calcinations in a muffle furnace at a temperature of 500°C and a period of 2 hours to make crystalline ZnO nanoparticles.In this way, high purity nanoparticles of ZnO with controlled morphology and phase composition could be produced, and their structure and optical properties could be further characterized.

Characterization Techniques
To evaluate the structural and optical properties of the synthesized ZnO nanoparticles, a combination of analytical techniques was employed, including X-ray diffraction (XRD), ultraviolet-visible (UV-Vis) spectroscopy, and Fourier-transform infrared (FT-IR) spectroscopy.

X-ray Diffraction (XRD)
The crystalline structure and the purity of the phases of ZnO nanoparticles were determined by X-ray diffraction analysis. The diffraction pattern of ZnO shows sharp peaks associated with crystalographic planes of (100), (002) and (101) of ZnO which are associated with the hexagonal wurtzite crystal structure. There were no secondary phases or impurities so the phase purity was high. Debye-Scherrer equation used to calculate average size of crystallites:
$$n\lambda = 2d \sin(\theta)$$
Where:
n is an integer (e.g., 1, 2, 3, …)
λ is the X-ray wavelength (for example, 1.54 Å for copper)
d is the spacing between layers of atoms
θ is the angle at which the X-ray hits the layer

The angle (θ) will allow us to get different d-values in the sample. This angle, which is measured by the XRD machine, and the intensity of the diffracted rays results in a unique pattern similar to a fingerprint that is unique to a substance. Almost 95% of solid

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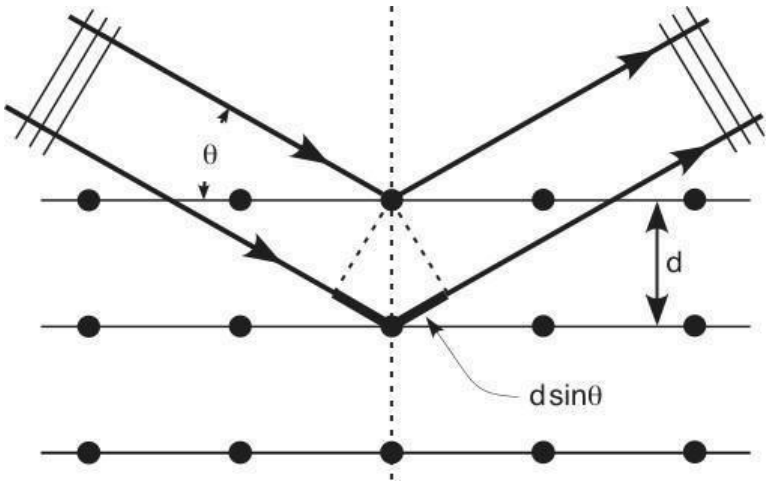
materials are crystalline, and each crystal has its own specific XRD pattern. Today, more than 50,000 inorganic and organic crystal patterns have been recorded and are used as reference data. XRD is not only used to identify materials, but also to determine structural details, such as the arrangement of atoms, interatomic distances, and bond angles. These are the reasons why XRD is a very important and powerful technique in materials science and solid-state chemistry.

Fig 1 Reflection of x-rays from two planes of atoms in a solid



Ultraviolet–Visible (UV-Vis) Spectroscopy

The optical properties of the ZnO nanoparticles were examined using UV–visible absorption spectroscopy. The absorption spectrum revealed a prominent peak at approximately 355 nm, along with an excitonic absorption feature near 258 nm, indicative of monodisperse nanoparticles with well-defined size distribution. The estimated optical band gap, derived from the absorption edge, was approximately 3.46 eV, aligning well with values reported for ZnO nanomaterials. These findings confirm the nanoscale dimensions and semiconducting behavior of the synthesized particles.

Fourier Transform Infrared (FT-IR) Spectroscopy

FT-IR spectroscopy was employed to investigate the chemical bonding and functional groups present in the ZnO nanoparticles. The spectrum exhibited distinct absorption bands around 457 cm^{-1} and 545 cm^{-1}, corresponding to Zn–O stretching vibrations, thereby confirming the formation of ZnO. Minor peaks were also observed in the range associated with atmospheric CO_2, likely due to ambient exposure during sample handling. The absence of significant organic or hydroxyl peaks indicates effective washing and thermal treatment of the nanoparticles.

Results and Discussion

X-Ray Diffraction (XRD) Analysis

The XRD pattern of the synthesized ZnO nanoparticles confirmed the formation of a pure crystalline phase with no evidence of secondary phases or impurity peaks. The

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diffraction peaks matched well with the standard JCPDS card for hexagonal wurtzite ZnO, indicating the successful synthesis of phase-pure material. The average crystallite size, calculated using the Debye–Scherrer formula, was found to be approximately 22.29 nm. A slight increase in crystallite size with increased sintering temperature was observed, consistent with crystal growth behavior during thermal treatment.

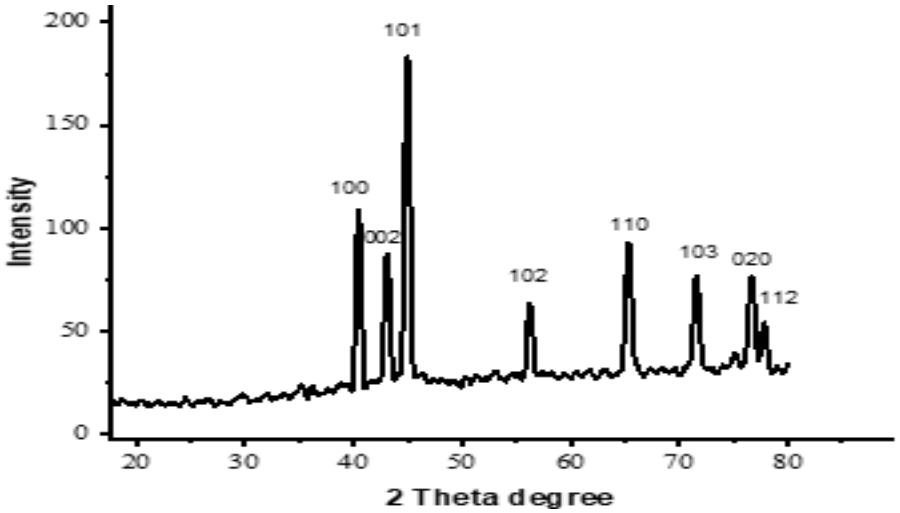

Figure 2 XRD spectrum of the zinc oxide nanoparticles

UV–Visible Spectroscopy Analysis

The UV–Vis absorption spectrum of the ZnO nanoparticles exhibited a sharp and intense absorption peak centered around 355 nm, along with a distinct excitonic absorption at approximately 258 nm. These features confirm the nanoscale dimensions of the synthesized particles and suggest a low degree of polydispersity. The calculated optical band gap was approximately 3.46 eV, which aligns well with values reported in the literature for ZnO nanoparticles. The sharpness of the absorption peak further indicates the uniformity and monodispersity of the particle distribution.

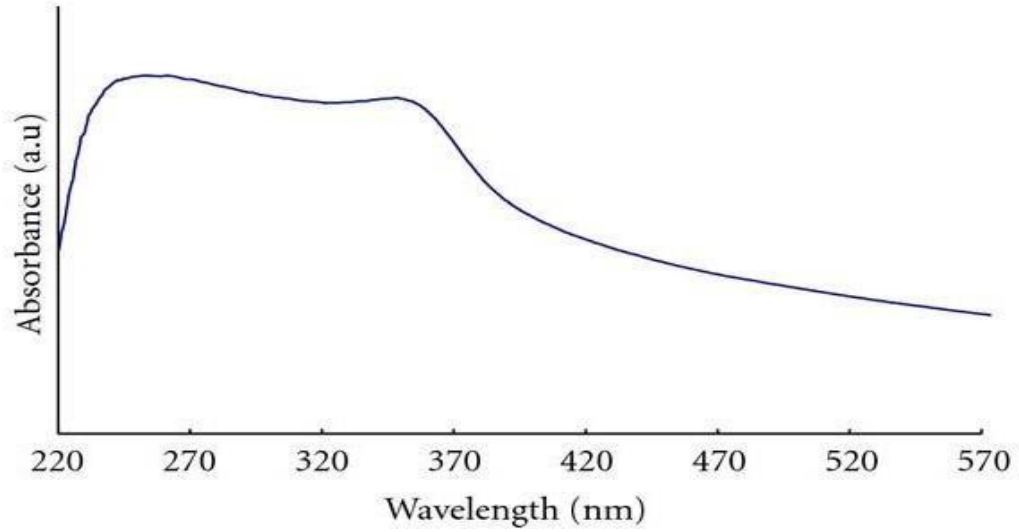

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Figure 3 UV-Vis spectrum of the zinc oxide nanoparticles

Fourier Transform Infrared (FT-IR) Spectroscopy Analysis

The FT-IR spectrum revealed characteristic Zn–O stretching vibrations at approximately 457 cm^{-1} and 545 cm^{-1}, confirming the formation of ZnO nanoparticles. Minor peaks attributed to residual CO_2 were also observed, likely due to atmospheric exposure. The absence of significant organic or hydroxyl functional groups validates the effectiveness of the washing and calcination processes in removing residual by-products and moisture.

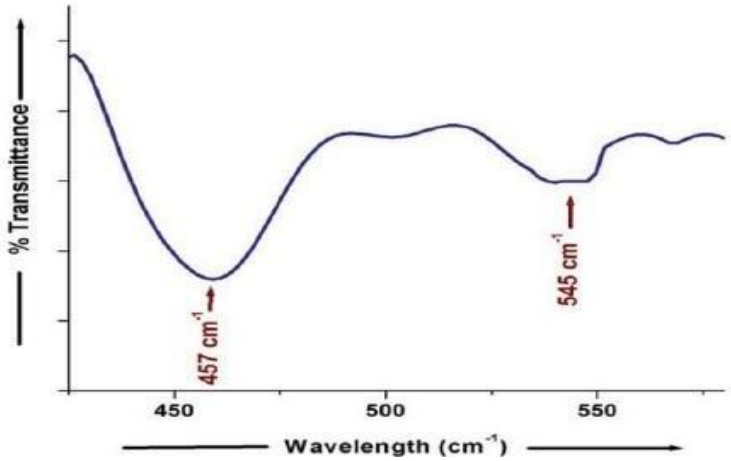

Figure 4 FT-IR spectrum of the zinc oxide nanoparticles

The results from all characterization techniques collectively confirm the successful synthesis of high-purity, nanosized ZnO particles with defined structural and optical properties. Additionally, the observed characteristics are consistent with those reported in previous studies, indirectly validating the influence of key synthesis parameters such as temperature and pH on particle morphology and crystallinity.

Conclusion

Zinc oxide (ZnO) nanoparticles were successfully synthesized using a simple, cost-effective co-precipitation method. Structural characterization via X-ray diffraction confirmed the formation of a pure hexagonal wurtzite phase, with average crystallite sizes ranging between 15 and 22 nm. Optical analysis using UV–visible spectroscopy revealed sharp absorption features indicative of monodisperse, nanoscale particles, while FT-IR spectra verified the presence of Zn–O bonding and the absence of significant impurities.

The synthesis route demonstrated excellent reproducibility and yielded nanoparticles with properties favorable for a wide range of applications, including electronics, photocatalysis, sensors, and biomedical fields. Moreover, the scalability, environmental

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friendliness, and control over particle characteristics highlight the potential of this method for industrial-level production of ZnO nanomaterials.

Future studies may focus on dopant incorporation and surface functionalization to further enhance the performance of ZnO nanoparticles in targeted applications.

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