



Synthesis of Zinc Oxide Nanoparticles via Sol-Gel Method, Characterisation, and Dielectric Analysis

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ABSTRACT

Zinc oxide (ZnO) nanoparticles were synthesised by a sol-gel/precipitation method. The synthesised ZnO nanoparticles were characterised by employing scanning electron microscopy (SEM), photoluminescence, and X-ray diffraction (XRD) techniques. Emission in the UV region was observed in the photoluminescence of the ZnO nanoparticles. Dielectric spectroscopy of the synthesised ZnO nanoparticle pellets was examined over a broad frequency range from 0.1 to 10^5 Hz. The capacitance of the ZnO nanoparticles decreases continuously with frequency because the dipoles receive less time to align in the field. The frequency variation of the dielectric loss of the ZnO pellets was studied. The electrical conductivity of the ZnO nanoparticles increases exponentially with frequency. The electrical conductivity and capacitance of ZnO are found to be strongly dependent on pellet thickness and on the frequency range, as revealed by the dielectric investigations.

1. INTRODUCTION

Most of the work in the field of nanomaterial synthesis deals with form, size, and composition. Each of these elements has a vital function in determining the properties of materials, leading to numerous technological applications [1]. ZnO nanoparticles are multifunctional materials with unique chemical and physical properties, such as high chemical stability, a high electrochemical coupling coefficient, a broad spectrum of radiation absorption, and excellent photostability [2, 3]. To obtain products with new properties, these materials must be modified on the nanometre scale [4]. Metal/metal-oxide nanostructures exhibit much higher catalytic activity than their bulk counterparts due to their comparatively small size and well-defined surface morphology; after reaction, they can be separated by centrifugation and recycled without loss of efficacy, and several noble-metal and metal-oxide nanostructures exhibit good catalytic activity in dye degradation [5]. The dynamic interplay between metal nanoparticles and oxide supports under redox conditions has also been discussed for related systems [6].

Zinc oxide is an n-type semiconductor with a wide band-gap energy (3.37 eV at room temperature) and a high excitation binding energy (60 meV). The large surface-to-volume ratio of ZnO nanostructures leads to interesting chemical and physical properties, and the surface of zinc oxide nanoparticles must be modified to improve dispersion. ZnO nanostructures can be synthesised using vapour–liquid–solid (VLS) synthesis [7]. The electrical properties of ZnO are strongly influenced by composition and structure [8]. Moreover, metal-oxide nanoparticles, namely zinc oxide (ZnO), indium oxide (In_2O_3), tin oxide (SnO_2),

and doped oxides, have attracted much attention recently; ZnO is of increasing interest due to its abundance, low cost, and non-toxicity relative to other materials [9]. Metal-oxide nanoparticles are important fillers for enhancing the reliability of electrical insulation and attaining compact design in power apparatus using polymer nanodielectrics [10]. Today, many firms apply nanotechnology in research, engineering, medicine, and other disciplines. Nanoparticles exhibit properties that vary with their size, quantum confinement, and surface plasmon resonance, and research over the last 20 years has focused on metal-oxide nanoparticles because of their unique electrical, optical, mechanical, magnetic, and chemical properties [11]. During the past few years, semiconductor compounds have attracted much attention for optics due to their innovative optical and transport characteristics; the wide-band-gap semiconductor ZnO is an excellent material for optical transparency and luminescence in the near-UV and visible range [12, 13]. Because many physicochemical preparation methods have a negative environmental impact, most researchers are now focused on eco-friendly approaches [14]. ZnO nanostructures are likewise of interest in biosensor applications [15], and green and mechanochemical routes can produce metal- and metal-oxide-based nanoparticles that act as catalysts in many industrial processes, whose formation mechanisms and redox processes are highly sensitive but specific [16]. Low-frequency dielectric properties and transverse optical phonon modes of single-crystal ZnO have also been investigated in earlier studies.

Beyond the materials themselves, the deployment of advanced technologies in research and industry also raises questions of algorithmic governance, justice, and the future of work [17].

In the present work, a sol-gel technique was used to synthesise zinc oxide nanoparticles. The morphology of the ZnO nanoparticles was evaluated using scanning electron microscopy (SEM), and the dielectric properties of the ZnO pellets were investigated for applications such as capacitance, dielectric loss, and electrical conductivity.

2. EXPERIMENTAL

2.1 Chemicals

In this research, ZnO nanostructures were synthesised by sol-gel technology using zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, $\geq 99\%$ purity), sodium hydroxide (NaOH , $\geq 98\%$, Sigma-Aldrich), ethanol ($\text{C}_2\text{H}_5\text{OH}$, HmbG Chemicals), and distilled water. Zinc acetate dihydrate acted as the precursor and ethanol as the reagent; the solvent medium was distilled water.

2.2 Preparation of Zinc Oxide (ZnO)

Zinc oxide nanostructures were prepared using a sol-gel technique. A sol was prepared by weighing 2 g of zinc acetate dihydrate and 8 g of sodium hydroxide on a weighing balance. Then 15 mL and 10 mL of distilled water were measured using a measuring cylinder. The 2 g of zinc acetate dihydrate was dissolved in 15 mL of distilled water, and the 8 g of sodium hydroxide was dissolved in 10 mL of distilled water. Each solution was stirred steadily for about five minutes. The zinc acetate solution was then added to the sodium hydroxide solution with continuous stirring on a magnetic stirrer for about five minutes. After that, 100 mL of ethanol was filled into a burette and titrated dropwise into the mixture. A white precipitate formed as a result of the reaction [18].

3. MEASUREMENTS

3.1 Characterisation Techniques

Room-temperature photoluminescence spectra of the ZnO nanopowder were recorded with an excitation wavelength of 320 nm. The morphology of the ZnO was characterised by SEM.

3.2 Dielectric Relaxation Spectra

Dielectric relaxation spectra (DRS) of the ZnO nanoparticle pellets were studied using a Hioki 3533 HiTester LCR meter with silver foil as a blocking electrode. The pellets were circular with a diameter of 1 cm and thicknesses of 1, 2, 3, 4, and 5 mm. The electrical conductivity (σ) was calculated from the dielectric values by the following equation:

$$\sigma_{AC} = \omega \varepsilon_0 \varepsilon' \tan \delta \quad (1)$$

where $\omega = 2\pi f$ (f is the frequency), ε_0 is the vacuum permittivity, and ε' is the dielectric permittivity (dielectric constant):

$$\varepsilon' = \frac{C_p}{C_0} \quad (2)$$

Electrical conductivity was therefore determined using the dielectric data [19].

4. RESULTS AND DISCUSSION

4.1 X-Ray Diffraction (XRD)

The XRD pattern of the zinc oxide crystallites was consistent with the Joint Committee on Powder Diffraction Standards (JCPDS) database card 36-1451. The diffraction peaks of the ZnO nanoparticles are shown in Figure 1. The quality of the starting material used in the manufacture of the zinc oxide nanoparticles accounts for the absence of additional peaks [20]. The high-intensity (101) peak was used to calculate the lattice parameters. The XRD peaks at 38.60° , 44.10° , 60.0° , 65.1° , and 70.48° were identified as the (101), (102), (110), (103), and (112) reflections, respectively.

The product has a fine crystalline structure, as demonstrated by the sharp and strong diffraction peaks. The average crystallite size (D) of the ZnO nanoparticles was estimated using Scherrer's formula:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (3)$$

where β is the full width at half maximum intensity of the diffraction peak, λ is the wavelength of Cu K α radiation (1.5418), and θ is the Bragg diffraction angle. The average crystallite size was found to be 41 nm; the larger apparent particle sizes of about 80 nm observed by SEM are attributed to agglomeration of the crystallites. The lattice parameters obtained from the resolved peaks may be used to quantify the crystallite size more accurately. The results are consistent with previously reported sol-gel-derived ZnO nanoparticles.

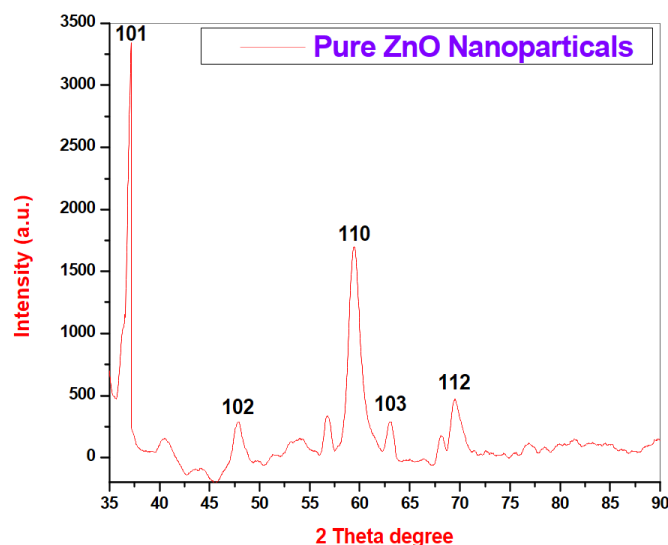


Figure 1. XRD spectrum of ZnO nanoparticles

4.2 Photoluminescence Spectra

When the dimensions of a semiconducting material are reduced to the nanometre scale ('quantum size effects'), its physical properties are altered; for example, the band-gap energy of ZnO increases by quantum confinement, which is observed in the photoluminescence [21]. The figure 2 shows the photoluminescence spectrum of the ZnO nanopowder excited at 320 nm at room temperature. The spectrum shows two emission peaks, one at 392 nm (UV region), which is the near-band-gap excitonic emission, and the other at 520 nm, which is attributed to singly ionised oxygen vacancies and originates from the radiative recombination of a photo-generated hole with an electron at an oxygen vacancy [22, 23]. The narrow luminescence peak (full width at half maximum of only a few nanometres) also indicates a narrow size distribution of the nanoparticles in the powder [24].

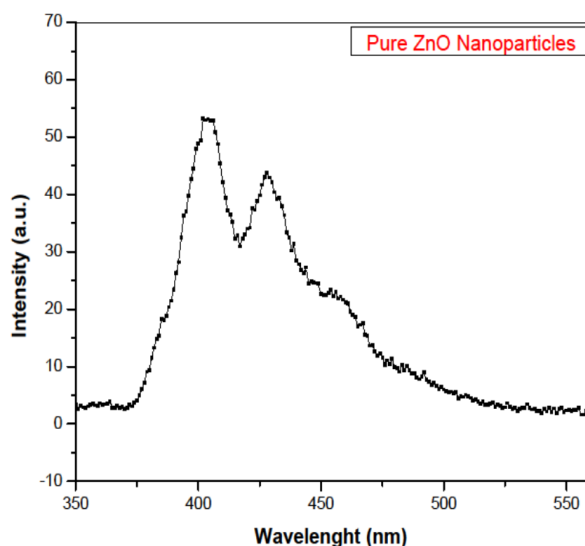


Figure 2. Photoluminescence spectra of ZnO nanoparticles

4.3 Scanning Electron Microscopy (SEM)

SEM employs a high-intensity electron beam to scan a material and produce surface characteristics, and it is used to detect the exterior morphology and size of the crystalline particles. As seen in the figure 3, a considerable fraction of the ZnO nanoparticles is spherical in shape, and small agglomerates are also transformed into bigger particles. The SEM photomicrograph shows individual ZnO nanoparticles and different aggregates. The apparent morphology of the ZnO nanoparticles is spherical with aggregated conditions at 25,000 $\times$ magnification. Variations in form, size, and spatial organisation of the particles may be due to variations in the synthesis process of the ZnO nanoparticles [25, 26].

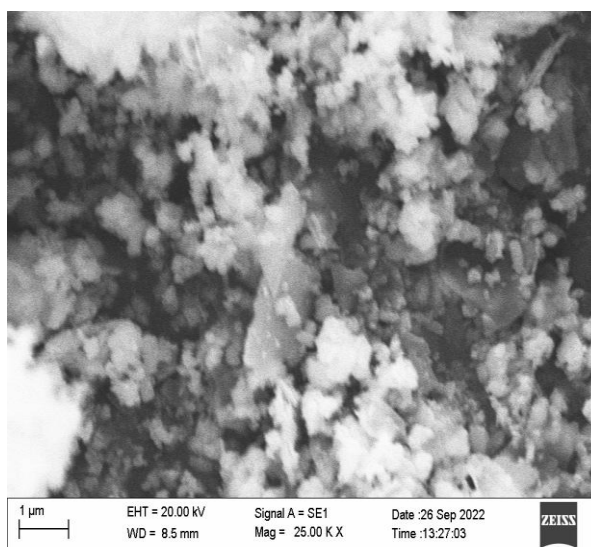


Figure 3. SEM image of ZnO nanoparticles

4.4 Capacitance (C)

Capacitance (C) is an important parameter for dielectric and energy-storage applications. The figure shows the variation of the capacitance of the prepared ZnO nanoparticle pellets of different thicknesses (1, 2, 3, 4, and 5 mm) with frequency. The capacitance is higher at low frequency and decreases continuously with increasing frequency, because the dipoles receive less time to align with the field. All types of polarisation (dipolar, interfacial, electronic, and ionic) contribute at low frequency, which results in the higher capacitance value. The figure 4 also shows that, within the measured range, the capacitance increases with the thickness of the ZnO pellets up to 5 mm, which can be connected with the increased resistance of the pellets [27, 28].

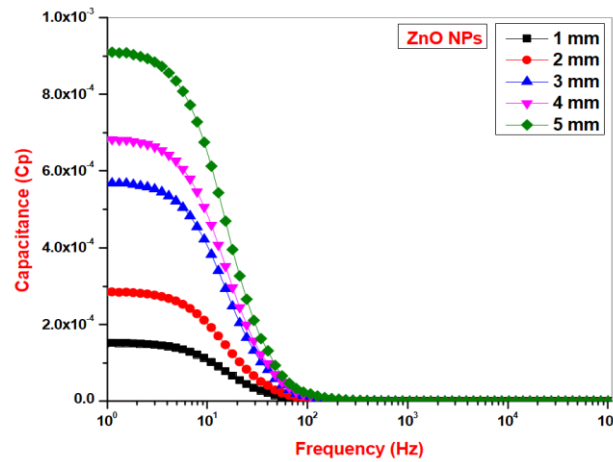


Figure 4. Capacitance (C) plots of zinc oxide (ZnO) nanoparticle pellets measured as a function of frequency

4.5 Dielectric Loss

The dielectric loss (ϵ'') of a material describes the energy dissipated during electrical polarisation. The figure 5 shows the variation of the dielectric loss with frequency for the different thicknesses (1–5 mm) of the produced ZnO agglomerated nanoparticles. The dielectric loss is greater at low frequency due to the presence of dipolar, interfacial, electronic, and ionic polarisation, and it decreases as the frequency increases. The figure also indicates that the value increases with the thickness of the ZnO pellets up to 5 mm within the measured range [29].

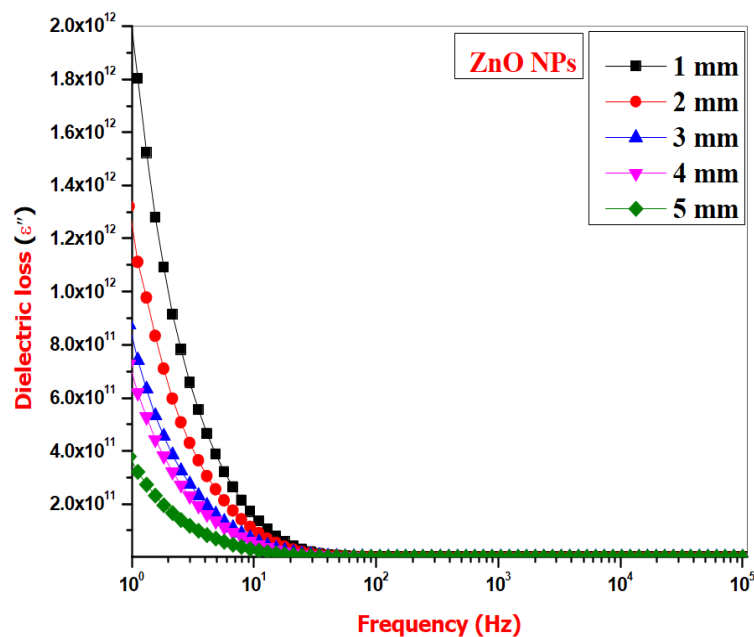


Figure 5. Dielectric loss plot of zinc oxide (ZnO) measured as a function of frequency: effect of thickness

4.6 Electrical Conductivity

The electrical conductivity (σ) quantifies how well a substance can transport electrical current (Figure 6). The frequency-dependent electrical conductivity of the ZnO nanoparticle pellets increases exponentially with increasing frequency, and within the measured range it increases with the thickness of the ZnO pellets up to 5 mm, consistent with the increase in dielectric permittivity [30, 31, 32].

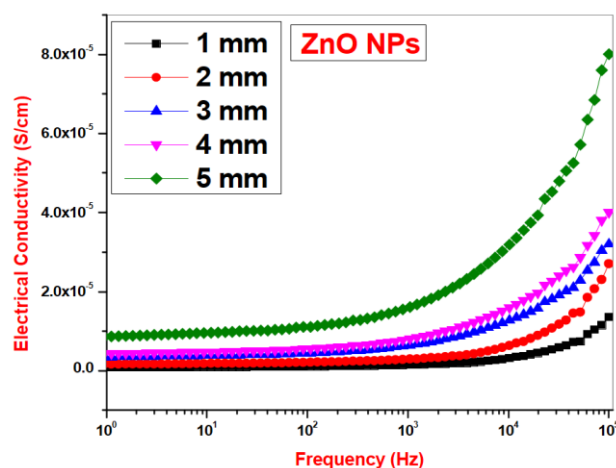


Figure 6. Electrical conductivity plot of zinc oxide (ZnO) measured as a function of frequency: effect of thickness.

5. CONCLUSIONS

The sol-gel method was successfully employed to synthesise zinc oxide (ZnO) nanoparticles. The photoluminescence spectrum of the ZnO nanopowder at an excitation wavelength of 320 nm and room temperature shows two emission peaks. The strong XRD diffraction peaks indicate that the material is crystalline, with an average crystallite size of about 41 nm from the Scherrer equation. The SEM image shows predominantly spherical and aggregated nanoparticles. The capacitance, dielectric loss, and electrical conductivity of the synthesised ZnO were investigated as functions of frequency and pellet thickness. The capacitance and dielectric loss are higher at low frequency and decrease with increasing frequency, whereas the electrical conductivity increases with frequency and with pellet thickness within the measured range. These behaviours may be attributed to charge-carrier activation and the formation of conduction channels through the pellet volume as the nanoparticles interact. The results indicate that the synthesised ZnO nanoparticles are suitable for dielectric and electronic applications.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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