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The Effect of Solvents Water, Ethanol, Methanol and Acetone on the Morphological and Optical Properties of ZnO Nanoparticles Synthesized by using Green Method

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ABSTRACT: Zinc oxide (ZnO) nanoparticles were produced using environmentally friendly and cost-effective methods, involving solvents with varying polarities. Throughout the experimentation, certain factors such as temperature, concentration, time, and pH were kept constant, while solvents like water, ethanol, methanol, and acetone were changed. The resulting particles underwent a series of characterization techniques, including X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infrared (FTIR) analysis, UV-visible absorption spectroscopy, and assessment of antibacterial properties. The XRD and SEM analyses confirmed ZnO nanoparticle formation. The size of the ZnO nanoparticles varied depending on the solvent used, with average crystallite sizes of 51.32nm, 81.94nm, 43.71nm, and 95.34nm achieved using water, ethanol, methanol, and acetone, respectively. The nanoparticles that were prepared demonstrated notable antibacterial activity against a range of microorganisms. It is noteworthy that the presence of hydroxyl groups in water and ethanol had a significant impact on the entire process, including the nucleation, growth, and termination of the nanoparticles. This interaction resulted in the formation of non-spherical particle shapes. From the analysis of the results, it is evident that the choice of solvents in this study played a critical role in determining the surface structure, structural characteristics, and optical properties of the ZnO nanoparticles.

Keywords: Zinc oxide, UV-visible, SEM, XRD, FTIR, nanoparticles.

INTRODUCTION

Semiconductors at the nanoscale have garnered significant attention owing to their exceptional properties (El-Sayed, 2004). One of the striking features of nanoparticles is their size-dependent electrical and optical behaviors, a phenomenon known as the quantum confinement effect (Pickett and O'Brien 2001; Landes *et al*., 2002). Zinc oxide (ZnO) stands out as one of the few semiconductors that prominently exhibit the quantum confinement effect. ZnO is characterized by a wide band gap (3.34 eV), and its versatility has led to applications across various fields (Fu *et al.*, 2007). Remarkably, it is an environmentally friendly oxide, recognized for its non-toxic nature and its capacity to absorb ultraviolet (UV) radiation. Due to these attributes, ZnO is employed as a UV absorbent in products such as sunscreens and plays an important role in solar energy conversion (Becheri *et al.*, 2008). Janjal *et al.* (2017) reported that zinc oxide nanoparticles were prepared by leaf extract of guava plant. Zinc oxide (ZnO) finds diverse applications, including photovoltaic devices, gas sensors, photocatalysis, transparent conducting coatings, and electrostatic transducers. Extensive documentation underscores the significant impact of material shape and size on its properties and applications (Moloto *et al.*, 2009; Jun *et al.*, 2006). Consequently, substantial efforts are being channeled into the control of particle size and shape. Nipane *et al*. (2012) obtained the ZnO-Nps of size about 50-100 nm. However, achieving this control remains a difficult challenge for synthetic chemists in the field of nanoscience. Various factors, such as time, temperature, concentration, precursors, capping agents, and solvents, among others, have been reported to exert influence on nanoparticle shape and size (Tang *et al.*, 2000; Yang *et al.*, 2000). ZnO stands out as a particularly fascinating metal oxide due to its ability to manifest a myriad of intricate morphologies (Sun *et al.*, 2008; Zhang, 2004). In this study, we investigate the influence of solvents on the morphology of ZnO nanoparticles. Solvents are pivotal in chemical reactions as they not only serve as reaction media but also help control reaction temperatures by establishing the maximum attainable temperature. Various methods have been employed for the synthesis of ZnO, including a novel sol-gel technique proposed by which yields stable zinc oxide colloids featuring nano-sized wurtzite crystals (Bahnemann *et al.*, 1987). Different types of techniques have been used to synthesize ZnO such as sputtering, spray pyrolysis Solvothermal Hydrothermal, Ball

milling method and Wet Chemical method. Jkun *et al*. (2006), Zhu *et al*. (2008) reported various hierarchical nanostructures of ZnO via microwave-assisted route. ZnO boasts an array of advantageous properties; it is non-toxic, possesses self-cleansing abilities, and is skin-friendly, antimicrobial, and dermatologically compatible. It is widely employed as a UV blocker in sunscreens and finds extensive use in numerous biomedical applications. Additionally, ZnO exhibits remarkable resistance to microorganisms. Several reports indicate strong antibacterial activity for CaO, MgO, and ZnO. This activity is attributed to the generation of reactive oxygen species on the surface of these oxides (Yang *et al.*, 2000).

MATERIALS AND METHODS

A. Materials

We purchased and made use of zinc acetate, sodium hydroxide, ethanol, acetone, and methanol without extra purification.

B. Method for Preparing the extract

Infusion is a method of extraction, equivalent to maceration. Aegle marmelos's fresh leaves are collected, cleaned with tap water, and then again cleaned with distilled water. After being cleaned, the leaves were dried in the shade and then ground into a fine powder using a mortar and pestle. To prepare the extract, fill four containers with 5 grams of fine powder each, then add 50 milliliters of a solvent (acetone, methanol, ethanol, or water), and let it soak for 24 hours. Next, filter paper Whatman No. 1 was used to filter the extract. The final extract was refrigerated at 4°C to allow for further processing.

C. Zinc Oxide Nanoparticle Synthesis

Zinc acetate and Aegle marmelos leaf extract have been combined with various solvents to yield the zinc oxide nanoparticles sustainably. Zinc acetate was dissolved in several solvents, such as water, ethanol, methanol, and acetone. After that, the mixture was continuously stirred for an hour using a magnetic stirrer to ensure that the zinc acetate was thoroughly dissolved. Leaf extract and sodium hydroxide solution were added, stirring constantly, drop by drop until the zinc acetate completely dissolved. Two hours were allowed to pass after the sodium hydroxide and extract were completely added to the process. Following the reaction's result, the solution was let to settle for the night, and the supernatant solution was then discarded carefully. After centrifuging the residual solution for ten minutes, the supernatant was discarded.

1. Leaves of Aegle marmelos; 2. Dried leaves; 3. Powder of dried leaves.

Fig. 1. Aegle marmelos leaves.

1. Extract in different solvents (Ethanol, Methanol, Acetone and Water); 2. Prepared ZnO Nanopowders in four solvents.

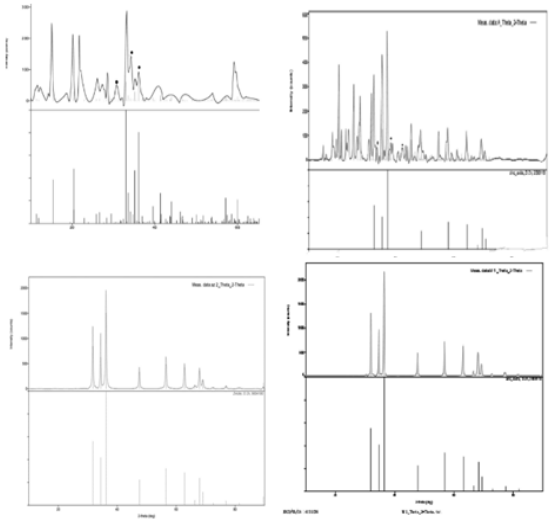
Fig. 2. Aegle marmelos leaves extract and prepared ZnO nanopowders.

The resulting nanoparticles were cleaned using purified water. To remove the byproducts that were attached to the nanoparticles, washing was carried out. The nanoparticles were cleaned and then dried for four hours at a maximum temperature of 70°C in a hot air oven.

RESULTS AND DISCUSSION

A. X-ray Diffraction

X-ray diffractometry has been used to check the purity and find the phase of the particles. The hexagonal phase ZnO has been given a value that corresponds to the XRD diffraction peaks. With the exception of the relative intensities based on due to their random orientation, the morphologies from ethanol and acetone show comparable diffraction patterns. Asterisks indicate the presence of impurities in ethanol and acetone, while the XRD pattern for water and methanol was indexed to pure hexagonal phase ZnO with no indication of zinc acetate. The unreacted zinc acetate is the source of contaminants. The Debye Scherer Formula is used to determine the average crystalline size of the particles. The samples' average crystallite sizes are 81.94 nm in ethanol solvent, 43.71 nm in methanol, 95.34 nm in acetone, and 51.32 nm in water. In comparison with ethanol and acetone, the average crystallite size for water and methanol was smaller (Khoza *et al.*, 2012).

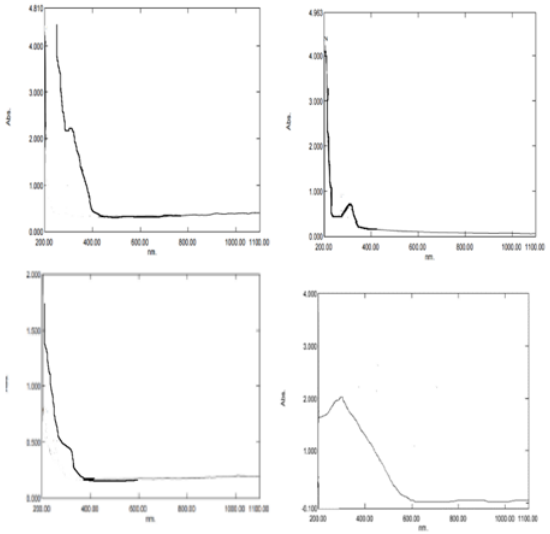


1. XRD ZnO NPs solvent ethanol; 2. XRD ZnO NPs solvent Acetone; 3. XRD ZnO NPs solvent Methanol; 4. XRD ZnO NPs solvent Water.

Fig. 3. XRD of ZnO NPs for different solvents

B. Analysis of Ultraviolet-Visible Spectra

UV-visible spectrophotometry was used to determine the maximum absorbance of the zinc oxide nanoparticles. Fig. 4 displays the UV-Vis absorption spectra of zinc oxide nanoparticles. The optical properties of the nanoparticles have been studied using ultraviolet and visible absorption spectroscopy throughout the 200–800 nm range. For several solvents, the biosynthesized materials showed strong absorption bands. ZnO nanoparticles in ethanol produce a peak at 332 nm, methanol produces a peak at 339 nm, acetone produces a peak at 330 nm, and water produces a peak at 358 nm when employed as a solvent. That the produced products are pure ZnO nanoparticles is confirmed by the spectra's absence of any other absorbance peak. The band gap and energy of ZnO nanoparticles produced using ethanol, methanol, acetone, and water as solvents were determined to be 3.73 eV, 3.65 eV, 3.75 eV, and 3.82 eV, respectively, based on the equation Eg = 1240/λeV. Variations in the average crystal size of the nanoparticles may be the reason for the variation in Eg for different solvents (Sun *et al.*, 2008).

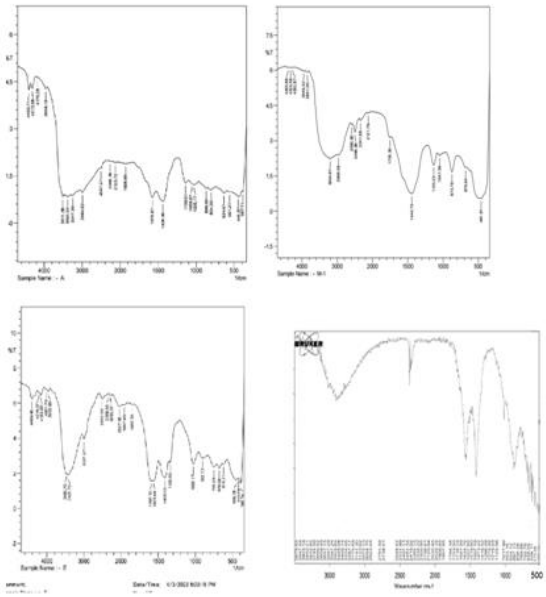


1. UV absorbence of ZnO NPs solvent ethanol; 2. UV absorbence of ZnO NPs solvent acetone; 3. UV absorbence of ZnO NPs solvent methanol; 4. UV absorbence of ZnO NPs solvent water

Fig. 4. UV absorbence of the biosynthesized ZnO nanoparticles in different solvents.

C. FTIR Spectral Analysis

The FTIR analysis of the four ZnO samples prepared using water, ethanol, methanol, and acetone are shown in Fig. 5. (1), (2), (3) and (4). The FTIR spectrum displayed the absorption band at 606.77 cm for ZnO NPs prepared by using acetone as solvent,456.18cm for ZnO NPs prepared by using methanol as solvent, 461.01cm for ZnO NPs prepared by using water as solvent and 551.31cm forZnO NPs prepared by using ethanol as solvent. This signifies the role of solvent in the preparation and formation of nanosized ZnO particles (Khoza *et al.*, 2012).

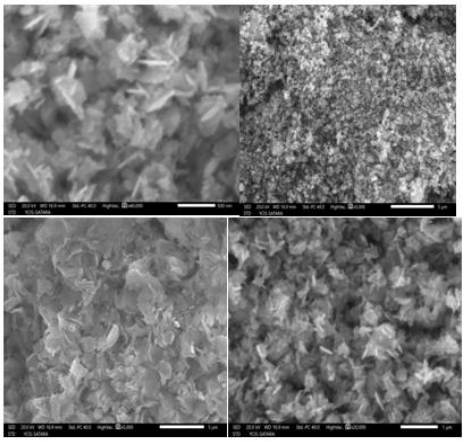


1. FTIR ZnO NPs solvent ethanol; 2. FTIR ZnO NPs solvent acetone; 3. FTIR ZnO NPs solvent methanol; 4. FTIR ZnO NPs solvent water

Fig. 5. Fourier transform infrared spectroscopy spectra of ZnO nanoparticles prepared by using different solvents.

D. Scanning Electron Microscopy

The investigation into the morphological characteristics and dimensions of the synthesized ZnO particles involved the utilization of scanning electron microscopy (SEM). As depicted in Fig. 6, the influence of different solvents (water, ethanol, methanol, acetone) on the particle morphology of ZnO nanopowders was examined across various magnifications. These images validate the successful formation of ZnO nanoparticles. The SEM micrographs of ZnO nanopowders prepared with ethanol, methanol, and acetone revealed an irregular spherical shape, with sizes falling within the nanometer range and exhibiting a narrow particle size distribution. Interestingly, the particles synthesized with water exhibited a distinctive flake-like shape. Upon closer inspection, the estimated particle size derived from SEM images ranged from 35 nm to 50 nm for samples synthesized with methanol and water, while those synthesized with ethanol and acetone showed a slightly larger size distribution ranging from 55 nm to 70 nm. The morphological features observed in the samples unveiled fine grains of ZnO that underwent a transformation into distinct particles upon scrutiny at varying magnifications. This comprehensive SEM analysis provides valuable insights into the influence of different solvents on the size and morphology of the synthesized ZnO nanoparticles, contributing to a deeper understanding of the synthesis process and its outcomes (Vanaja and Rao 2015).



1.ethanol; 2. Water; 3 methanol; 4. acetone.

Fig. 6. SEM images of ZnO nanoparticles prepared by green method.

E. Antibacterial Activity

The assessment of the antibacterial efficacy of the synthesized ZnO nanoparticles was conducted against pathogenic microbes, revealing noteworthy results. Among the tested microorganisms, Staphylococcus aureus exhibited the highest zone of inhibition, indicating a robust antibacterial effect. Conversely, Escherichia coli displayed the lowest zone of inhibition, suggesting a comparatively lesser impact on this particular bacterium. Detailed data on the zones of inhibition generated by ZnO nanoparticles are presented in Table 1 (Janaki *et al.*, 2015).

Table 1: Antibacterial activity for different solvents.

Plant species	Solvent	Zone of inhibition(mm)	
		E. coli	***S. aureus***
Aegle marmelos leaves	Water	0 mm	10 mm
	Ethanol	2 mm	5 mm
	Methanol	3 mm	9 mm
	Acetone	0 mm	8 mm

CONCLUSIONS

In this study, we successfully synthesized ZnO nanoparticles using four distinct solvents: ethanol, methanol, acetone, and water. The structural characterization revealed the crystalline nature of ZnO nanoparticles in the wurtzite phase. Notably, ZnO nanoparticles synthesized with water and methanol demonstrated a smaller crystallite size compared to those synthesized with ethanol and acetone. The UV-visible characterization indicated that the choice of solvent had minimal impact on the band gap, refractive index, and electron polarizability of ZnO nanoparticles. This suggests that the optical properties of ZnO nanoparticles remain relatively unaffected by the solvent used in the synthesis process. Our findings highlight the potential utility of ZnO nanoparticles in various optical devices.

FUTURE SCOPE

Biosynthesis emerges as a promising and environmentally benign method for ZnO nanoparticle production, representing an avenue for further exploration. The unique qualities of ZnO nanoparticles, encompassing their optical, physical, and antibacterial attributes, position them as valuable components in medical applications, cosmetics and diverse industries.

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Conflict of Interest. None.

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