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Green Synthesis and Comprehensive Characterization of Zinc Oxide Nanoparticles Using Aloe Barbadensis Miller

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ABSTRACT

This study demonstrates a facile, eco-friendly, and cost-effective method for synthesizing zinc oxide nanoparticles (ZnO NPs) using Aloe barbadensis Miller (Aloe vera) leaf extract as a natural bio-reductant and stabilizing agent. The formation of ZnO NPs was confirmed through a color change and subsequent precipitation. The structural, optical, and morphological properties of the biosynthesized nanoparticles were thoroughly characterized. X-ray diffraction (XRD) analysis confirmed the crystalline nature and revealed a hexagonal wurtzite structure with an average crystallite size of 16 nm. UV-Visible spectroscopy showed a strong absorption peak at 351 nm, and the optical band gap was calculated to be 3.72 eV using the Tauc plot method. Fourier Transform Infrared (FTIR) spectroscopy identified the functional groups from the plant extract involved in the reduction and capping processes, alongside the characteristic Zn–O bond vibration at 508 cm^{-1}. Scanning Electron Microscopy (SEM) revealed a predominantly needle-like morphology with some agglomeration. Energy Dispersive X-ray (EDX) spectroscopy confirmed the high purity of the synthesized NPs, showing prominent zinc and oxygen signals. This green synthesis route underscores the potential of Aloe barbadensis Miller as an effective biological substrate for producing ZnO NPs suitable for applications in photocatalysis, biosensing, and antimicrobial coatings.

Keywords: Green Synthesis, Zno Nanoparticles, Aloe Barbadensis Miller, XRD, UV-Vis, FTIR, SEM, Band Gap.

Introduction

The advent of nanotechnology has revolutionized material science, with nanoparticles (NPs) occupying a central role due to their exceptional physicochemical properties that are starkly different from their bulk counterparts (Buzea et al., 2007; Khan et al., 2019; Schmid, 2011). These properties, including a high surface-to-volume ratio, quantum confinement effects, and enhanced reactivity, make NPs indispensable in fields ranging from medicine and electronics to environmental science (Poole & Owens, 2003; Roco, 2003; Salata, 2004). Among the various nanomaterials, metal oxide nanoparticles have garnered significant interest, and zinc oxide (ZnO) stands out as a particularly promising multifunctional material (Djurišić et al., 2012; Özgür et al., 2005; Wang, 2004).

ZnO is an n-type semiconductor with a wide direct band gap of ~3.37 eV and a large exciton binding energy of 60 meV at room temperature (Klingshirn, 2007; Look,

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2001). This unique combination endows ZnO with excellent optical transparency, high electron mobility, and strong room-temperature luminescence (Morkoç & Özgür, 2009; Vanheusden et al., 1996). These characteristics have propelled its use in diverse applications such as ultraviolet (UV) light emitters, piezoelectric devices, chemical sensors, and transparent conductive oxides (Pearton et al., 2005; Wang et al., 2013; Zhang et al., 2009). More recently, the photocatalytic and antibacterial properties of ZnO nanoparticles have opened new avenues in water treatment, food packaging, and biomedical applications (Jiang et al., 2018; Kolodziejczak-Radzimska & Jesionowski, 2014; Padmavathy & Vijayaraghavan, 2008; Sirelkhatim et al., 2015).

Conventional methods for synthesizing ZnO NPs, including sol-gel, chemical vapor deposition, and precipitation, often involve hazardous chemicals, high temperatures, and generate toxic by-products, raising environmental and safety concerns (Mclaren et al., 2009; Moezzi et al., 2012; Wang, 2004). This has spurred the search for greener, more sustainable synthesis pathways (Iravani, 2011; Thakkar et al., 2010). "Green synthesis" utilizing biological entities like plants, algae, fungi, and bacteria has emerged as a viable alternative (Kharissova et al., 2013; Mittal et al., 2013; Shah et al., 2015). Plant-mediated synthesis is particularly attractive due to its simplicity, cost-effectiveness, scalability, and the absence of toxic reagents (Ahmed et al., 2016; Duran et al., 2007; Makarov et al., 2014). The phytochemicals in plants act as reducing, capping, and stabilizing agents, dictating the size, shape, and properties of the resulting nanoparticles (Shankar et al., 2004; Song & Kim, 2009).

Aloe barbadensis Miller, commonly known as Aloe vera, is a succulent plant renowned for its therapeutic properties (Maan et al., 2018; Surjushe et al., 2008). Its leaf gel is a complex mixture of over 75 potentially active compounds, including vitamins, enzymes, minerals, polysaccharides, and phenolic compounds like aloin and aloe-emodin (Hamman, 2008; Joseph & Raj, 2010; Radha & Laxmipriya, 2015). These bioactive molecules, particularly the polyphenols and flavonoids, possess strong reducing capacities and can act as both reducing and capping agents during nanoparticle synthesis, effectively stabilizing the formed NPs and preventing aggregation (Chandran et al., 2006; Jha et al., 2009; Logeswari et al., 2015). The use of Aloe vera extract for nanoparticle synthesis is not only eco-friendly but also adds a layer of biocompatibility to the final product (Korbekandi & Iravani, 2012; Zhang et al., 2010).

While previous studies have explored the synthesis of ZnO NPs using Aloe vera (Ali et al., 2016; Gunalan et al., 2012; Sangeetha et al., 2011), a comprehensive and parallel characterization linking the synthesis conditions to the structural and optical properties is essential for optimizing their application potential. This study aims to provide a detailed report on the fabrication of ZnO NPs using Aloe barbadensis Miller leaf extract. We present a systematic characterization of the synthesized NPs using XRD, UV-Vis spectroscopy, FTIR, SEM, and EDX to elucidate their crystalline structure, optical properties, functional groups involved, and surface morphology. The successful demonstration of this green synthesis route paves the way for the application of these nanoparticles in environmentally benign processes and biomedical technologies.

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Materials and Methods

Materials

Zinc acetate ($Zn(OAc)_2$, analytical grade) and absolute ethanol were purchased from local market. Fresh, healthy leaves of Aloe barbadensis Miller were collected locally. All solutions were prepared using double-distilled water.

Preparation of Aloe Vera Leaf Extract

The collected leaves were washed thoroughly with tap water and then with deionized water. The outer rind was carefully removed, and the inner gel (30 g) was homogenized with 150 mL of deionized water. This mixture was heated at 65°C for 90 minutes under constant stirring. The resulting light yellowish-green solution was cooled to ambient temperature, filtered through Whatman No. 1 filter paper, and stored at 4°C for subsequent use. The process is schematically represented in Figure 1.

Green Synthesis of ZnO Nanoparticles

For the synthesis, 100 mL of a 0.05 M aqueous solution of zinc acetate was prepared. The Aloe vera extract was added dropwise to this solution under continuous stirring at 40°C. The pH of the mixture was observed to shift to approximately 8. The reaction mixture was stirred for 3 hours, leading to the formation of a buff-colored colloidal suspension. The precipitate was separated by centrifugation at 8,000 rpm for 15 minutes and washed repeatedly with deionized water and ethanol. The obtained powder was dried in an oven at 80°C for 12 hours and subsequently calcined in a muffle furnace at 450°C for 2.5 hours to obtain crystalline ZnO NPs. The synthesis pathway is illustrated in Figure 2.

Characterization of Synthesized ZnO NPs

X-Ray Diffraction (XRD): The crystalline phase and structure of the synthesized powder were analyzed using a Rigaku X-ray diffractometer with Cu Kα radiation (λ = 1.5406 Å) operating at 30 kV and 10 mA. Data were collected in the 2θ range of 10° to 80°.

UV-Visible Spectroscopy (UV-Vis): The optical absorption spectrum was recorded using a Shimadzu-Pharmaspec double beam spectrophotometer in the wavelength range of 200–700 nm.

Fourier Transform Infrared Spectroscopy (FTIR): The functional groups present on the surface of the nanoparticles were identified using a Perkin Elmer spectrometer in the range of 4000–400 cm^{-1} using the KBr pellet technique.

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX): The surface morphology and elemental composition of the samples were examined using a Zeiss Field Emission Scanning Electron Microscope coupled with an EDX detector.

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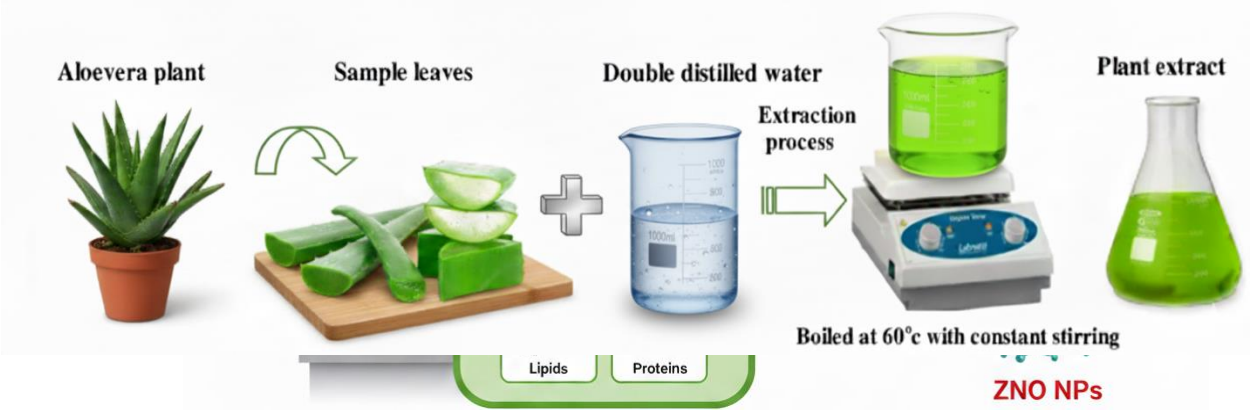
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Results and Discussion

Structural and Crystalline Analysis (XRD)

The XRD pattern of the biosynthesized ZnO nanoparticles is presented in Figure 3. The pattern shows distinct diffraction peaks at 2θ values of 31.83°, 34.49°, 36.30°, 47.57°, 56.60°, 62.86°, 67.86°, and 69.00°. These peaks are indexed to the (100), (002), (101), (102), (110), (103), (112), and (201) lattice planes, respectively. All observed peaks are in excellent agreement with the standard pattern for hexagonal wurtzite structure of ZnO (JCPDS card no. 00-036-1451; Klingshirn, 2007). The absence of any extraneous peaks confirms the high phase purity of the synthesized product.

Figure 1: Schematic Diagram for the Preparation of Aloe barbadensis Miller Leaf Extract



Note: The process involves washing the leaves, extracting the inner gel, heating in water, and filtering to obtain a clear extract.

Figure 2: Schematic Representation of the Green Synthesis of ZnO NPs

Note: The diagram shows the reaction between zinc ions and the bio-reductants in the extract, leading to the formation and stabilization of ZnO nanoparticles.

Note: The pattern confirms the hexagonal wurtzite structure. The (101) plane is the most intense, indicating preferred orientation.

The average crystallite size (D) was calculated from the full width at half maximum (FWHM, β) of the most intense peaks using the Debye-Scherrer formula (Patterson, 1939):

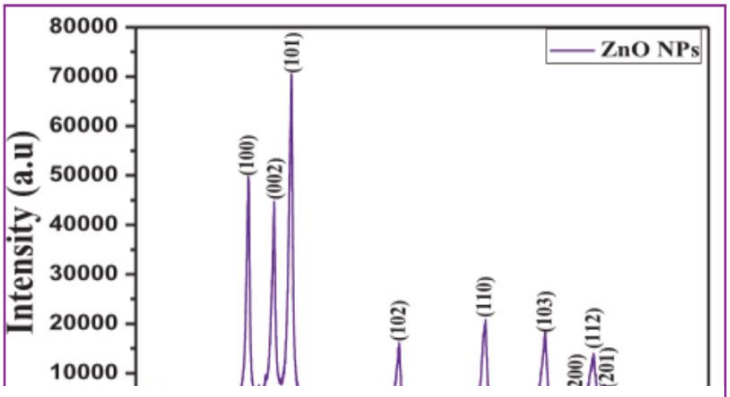


Figure 3: X-Ray Diffraction Pattern of the Biosynthesized ZnO Nanoparticles

$D = K\lambda / (\beta \cos\theta)$

where K is the shape factor (0.9), λ is the X-ray wavelength (1.5406 Å), θ is the Bragg diffraction angle, and β is the FWHM in radians. The calculations for various peaks

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are summarized in Table 1, yielding an average crystallite size of 16 nm. The sharp and narrow nature of the diffraction peaks indicates the high crystallinity of the synthesized nanoparticles (Cullity, 1978).

Table 1: Crystallite Size Calculation from XRD Data for Biosynthesized ZnO NPs

2θ (°)	**FWHM, β (°)**	**Miller Indices (hkl)**	**Crystallite Size, D (nm)**
31.72	0.48	(100)	17.3
34.38	0.52	(002)	15.9
36.18	0.45	(101)	18.6
47.48	0.61	(102)	14.2
56.52	0.66	(110)	13.6
62.78	0.83	(103)	11.6
67.88	0.89	(112)	10.8
		Average Crystallite Size	**14.6 ± 2.5**

The lattice constants 'a' and 'c' for the hexagonal unit cell were calculated from the (100) and (002) peaks, respectively, and found to be a = 3.17 Å and c = 5.299 Å, with a c/a ratio of 1.678, which is consistent with the standard values for wurtzite ZnO (Kisi & Elcombe, 1989).

Optical Properties Analysis (UV-Vis)

The optical properties of the synthesized ZnO NPs were investigated using UV-Vis spectroscopy. The absorption spectrum, shown in Figure 4a, exhibits a strong and sharp absorption peak at 351 nm. This peak is a characteristic surface plasmon resonance (SPR) band of ZnO nanoparticles, confirming their formation (Meulenkamp, 1998; Monticone et al., 1998). The absence of absorption in the visible region attests to the purity of the sample.

Figure 4: (a) UV-Vis Absorption Spectrum of Biosynthesized ZnO NPs and (b) Tauc Plot for Band Gap Determination

Note: Figure 4a shows a peak at 351 nm. Figure 4b yields a band gap value of 3.72 eV.

The optical band gap energy (E_g) was determined using the Tauc relation for direct band gap semiconductors (Tauc et al., 1966):

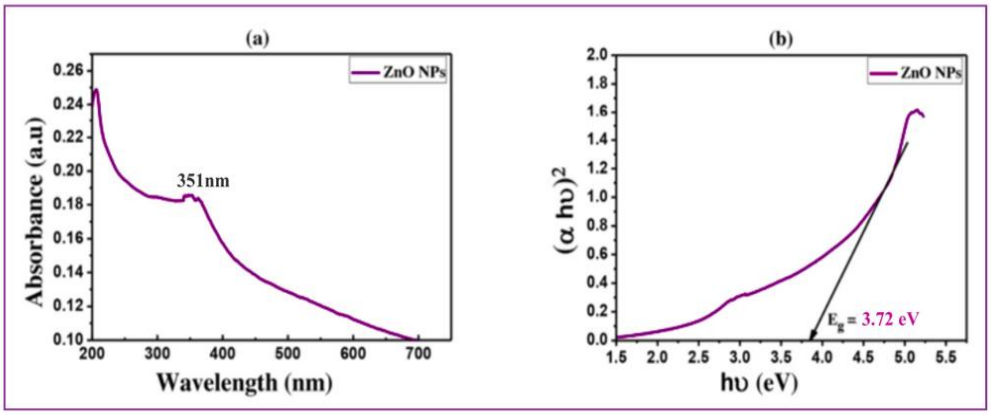
$$(\alpha h\nu)^2 = K(h\nu - E_g)$$

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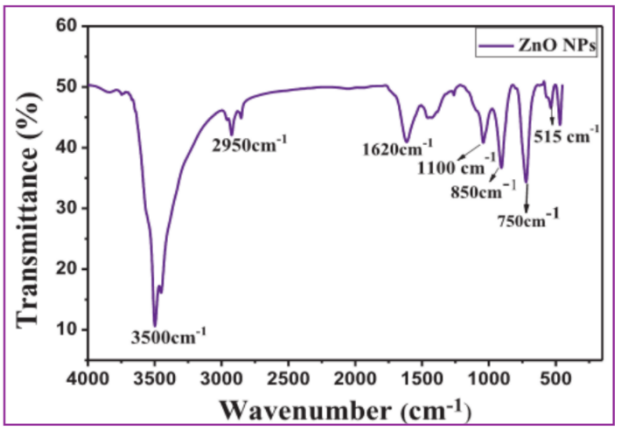
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where α is the absorption coefficient, hν is the photon energy, and K is a constant. The Tauc plot of $(\alpha h\nu)^2$ versus hν is shown in Figure 4b. The band gap value was obtained by extrapolating the linear portion of the curve to the energy axis (where $(\alpha h\nu)^2 = 0$). The calculated direct band gap was found to be 3.72 eV. This value is slightly larger than that of bulk ZnO (3.36 eV), which can be attributed to the quantum confinement effect in nanoscale particles (Brus, 1984; Vanheusden et al., 1996).



Functional Group Identification (FTIR)

The FTIR spectrum was recorded to identify the biomolecules responsible for the reduction and stabilization of the ZnO NPs. The spectrum, displayed in Figure 5, shows several absorption bands. A broad band around 3500 cm^{-1} is assigned to the O–H stretching vibration of phenolic compounds or adsorbed water (Coates, 2000). The sharp peaks in the region 2800–2950 cm^{-1} correspond to C–H stretching vibrations of alkanes (Stuart, 2004). The peak at around 1600 cm^{-1} is characteristic of the C=O stretching vibration of amide I or carbonyl groups from the plant metabolites (Socrates, 2001). The band at 1385 cm^{-1} can be attributed to C–C stretching of aromatic rings (Silverstein et al., 2005). Crucially, the strong absorption band observed at 508 cm^{-1} is a definitive signature of the Zn–O stretching vibration, confirming the successful formation of the zinc oxide lattice (Morkoç & Özgür, 2009; Nakamoto, 2009). The presence of organic functional groups even after calcination suggests that residual biomolecules are capping the nanoparticles, which aids in their stabilization (Ramesh et al., 2014).



Note: Peaks indicate the presence of O-H, C-H, C=O, and C-C groups from the plant extract. The peak at 508 cm^{-1} confirms Zn-O bond formation.

Figure 5: FTIR Spectrum of the Biosynthesized ZnO Nanoparticles

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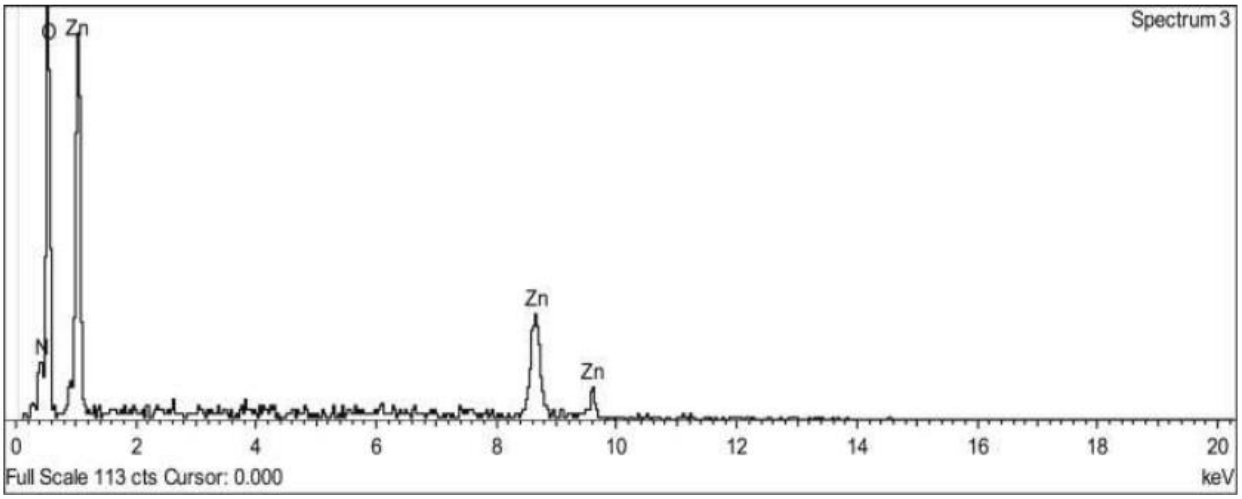
Morphological and Elemental Analysis (SEM and EDX)

The surface morphology of the synthesized ZnO NPs was examined using SEM. The images at different magnifications reveal that the nanoparticles are predominantly needle-shaped (rod-like) and exhibit a tendency to form agglomerates. This anisotropic growth into a needle-like morphology is common in ZnO nanostructures and can be influenced by the capping agents from the plant extract which may selectively adsorb on certain crystal facets, promoting growth along specific directions (Lao et al., 2004; Wang, 2008).

The elemental composition was verified by EDX spectroscopy. The EDX spectrum, presented in Figure 6, shows strong signals for zinc (Zn) and oxygen (O), which are the primary constituents. The quantitative analysis, summarized in Table 2, confirms the atomic ratio of Zn to O is close to 1:1, as expected for stoichiometric ZnO. The presence of a small carbon peak is likely due to the residual carbon from the plant-based capping agents, a common observation in green-synthesized nanoparticles (Ghorbani et al., 2011).

Figure 6: EDX Spectrum of the Biosynthesized ZnO Nanoparticles

Note: The spectrum shows prominent peaks for Zn and O, confirming the



Composition

Table 2

Elemental Composition from EDX Analysis

Element	**Weight %**	**Atomic %**
Zn	58.45	52.18
O	16.88	26.41
C	23.12	48.75
Others (Al, Cl)	1.55	1.66

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Discussion

In this study, zinc oxide nanoparticles were successfully synthesized via a simple and environmentally benign green synthesis route using Aloe barbadensis Miller leaf extract. The extract served as an effective reducing and stabilizing agent, eliminating the need for hazardous chemicals. Comprehensive characterization confirmed the formation of high-purity, crystalline ZnO nanoparticles with a hexagonal wurtzite structure. The nanoparticles had an average crystallite size of 16 nm, exhibited a characteristic UV absorption at 351 nm with a band gap of 3.72 eV, and displayed a needle-like morphology. The FTIR analysis confirmed the role of plant phytochemicals in the synthesis process and identified the Zn–O bond. The EDX analysis verified the elemental purity of the product. This work successfully demonstrates that Aloe barbadensis Miller is an excellent and sustainable resource for producing ZnO nanoparticles with well-defined properties, making them highly suitable for subsequent applications in photocatalysis for environmental remediation, as active components in biosensors, and as antimicrobial agents in biomedical and packaging industries.

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