Synthesis And Characterization Of Zinc Oxide Nanoparticles Using Green Synthesis Method


1, 3, 4 Research Centre in Electronic Science and Department of Electronic Science, Loknete Vyankatrao Hiray Arts, Science and Commerce College Panchavati, Nashik, Affiliated to SPPU, Pune, Maharashtra - 422003, India.

2 Department of Electronic Science, VADP’s Arts, Science & Commerce College, Shirsondi, Tal – Malegaon, Dist-Nashik, Affiliated to SPPU, Pune, Maharashtra – 423208, India.

3 Department of Physics, MGV’s, Arts, Science and Commerce College, Manmad, Dist. Nashik 423104, India

4 Department of Physics, MGV’s, Arts, Science and Commerce College, Surgana, Dist. Nashik 422211, India

Abstract: Nanoparticles (NPs) exhibit distinct features from their bulk materials, and they have applications in many different domains in life science. Green-synthesized NPs have attracted a lot of attention because of their intrinsic characteristics like efficiency, eco-friendliness, and cost-effectiveness. The current research focuses on the green synthesis of zinc oxide (ZnO) nanoparticles (NPs) employing wheatgrass extract. Wheatgrass extract acts as a capping agent, as evidenced by its presence on the surface of ZnO nanoparticles. X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Analysis (EDAX), and Fourier Transform Infrared spectroscopy (FTIR) were used to characterise the synthesised ZnO nanoparticles. The wurtzite hexagonal structure of the synthesised ZnO nanoparticles was polycrystalline in nature, with a preferential orientation along the (101) plane. Using Scherer's formula, the crystallite size was determined to be 36.18 nm. The morphological and elemental analysis of the films were investigated using SEM and EDAX tools, respectively. The presence of this extract on the surface of ZnO NPs was investigated using FTIR analysis, which confirmed the presence of many functional groups on the surface of ZnO NPs.

Keywords - ZnO nanoparticles, Wheatgrass extract, FTIR, Green-synthesis, Crystallite Size.
1. Introduction:

Environmental engineering is an interesting research topic that aims to improve human living standard. Environmental engineering uses nanotechnology in a variety of ways. Green eco-friendly approach has been impressively explored for the synthesis of a diverse range of nanostructures during the previous few decades [1]. Plant-based nanoparticles have numerous benefits over traditional physico-chemical processes and have a wide range of applications in biology and medicine [2].

ZnO is a rare inorganic compound found in nature. It's usually found as a crystalline substance. Manganese impurities in naturally occurring ZnO give it a classic red or orange color. ZnO is a white crystalline powder that is almost water insoluble when purified. The semiconductor nature of ZnO is n-type. It has a wide band gap of 3.37eV at ambient temperature with a high excitation binding energy (60meV), superior chemical stability, low dielectric constant, strong electrochemical coupling coefficient, and excellent optical absorbance [3].

ZnO NPs have been extensively applied in textile materials, beauty products, diagnostic, solar cells, sensing applications, dielectric materials, electric actuators, as a piezoelectric element, for room temperature UV lasing are only a few of the applications and even micro-electronics due to the relatively low toxicity and size dependant characteristics [3-6]. For biosynthesis of NPs for therapeutic reasons, ZnO has been proven to be more beneficial and efficient than other metals. Several investigations have shown that plant extracts can be used to synthesise ZnO NPs [4, 7]. The size of NPs is also affected by the plant type or source species from which the plant extract used for NP synthesis is derived [2, 5].

Agropyron repens, Brotedel Trigo, Agopyre, Doggrass, Elymus repens, Graminis Rhizoma, Quackgrass, Scotch Quelch, Triticum repens, and Wheat Grass were some of the other names for wheatgrass. This plant grows from the young grass of the ordinary wheat plant, Triticum aestivum. Wheatgrass juice and powder have numerous medicinal qualities for both humans and other animals [1, 8]. This plant's medicinal qualities include lowering blood pressure and cholesterol, preventing tooth decay, healing wounds, and acting as an antibiotic. Vitamin C, vitamin E, thiamin, niacin, pantothenic acid, protein, riboflavin, polyphenol, amino acid, and other substances were found in wheatgrass extract [9]. Although numerous investigations on nanoparticle syntheses have been conducted.

The incorporation of biological components in the nanoparticle synthesis always has been a preferred option for a green synthesis method. Green synthesis has been shown to be particularly effective at controlling the desired NP size and shape, in addition to the environmental ecosystem benefits [10].

2. Experimental Work:

2.1 Synthesis of ZnO nanoparticles using green synthesis method

In the current research work, commercially available AR grade (99.99 % purity) chemicals were used. ZnO nanoparticles have been synthesized by using Zinc nitrate hexahydrate Zn(NO₃)₂·6H₂O as a source of Zn. 20 gm of wheat grass pieces were kept in 100 ml distilled water and then boiled for 10 min. at 60 °C temperature. After that, 3 gm Zinc nitrate hexahydrate added in the prepared solution and stirred continuously for 2 hrs using magnetic stirrer up till the coloured of solution become yellow. The obtained...
precipitate was annealed at 600°C for 2 hours using muffle furnace. After that nanoparticles of ZnO obtained.

### 2.2 Structural Characterization:

The synthesized ZnO NPs were characterized by XRD, FESEM, and EDAX to study the structural properties, surface morphology, and elemental composition analysis respectively. Using FTIR analysis, the functional groups of ZnO NPs were confirmed.

#### 2.2.1 X-Ray Diffraction (XRD)

On a Rigaku diffractometer (DMAX-500), an X-ray diffractometer with CuKα radiation and wavelength (λ) = 0.154059 nm, XRD patterns of synthesized ZnO NPs were observed. The 2θ values obtained were compared to data files from the Joint Committee on Powder Diffraction Standards (JCPDS). Debye Scherer's formula, Eq. 1, was used to determine the crystallite size [3].

\[ D = \frac{K\lambda}{\beta \cos \theta} \]  

Where, 
D = Crystallite size,  
K = Scherrer constant (0.9),  
\( \beta \) = Full width of half maxima (FWHM),  
\( \lambda \) = wavelength of X source (0.154059 nm).

By using Bragg’s formula Eq.2, the interplaner spacing was calculated.

\[ d = \frac{\lambda}{2 \sin \theta} \]  

Where, \( \lambda \) = wavelength of X-radiation.

#### 2.2.2 SEM

The surface morphology of synthesized ZnO NPs were characterized using a Field Emission Scanning Electron Microscope (FESEM) [Model JOEL 6300 LA GERMANY].

#### 2.2.3 EDAX

Energy dispersive X-ray spectrometer (JOEL-2300, Germany) was used to study the elemental analysis of synthesized ZnO NPs.

#### 2.2.4 FTIR

Bonding characteristics of the thick films were analysed by using Shimadzu IR Affinity-1 Fourier transformed infrared spectrometer (FTIR). The FTIR was recorded from 400 to 4000 cm\(^{-1}\) with a resolution of 2 cm\(^{-1}\). FTIR analysis was used to evaluate the existence of functional groups of synthesized ZnO NPs.
3. Results and Discussion:

3.1 X-Ray Diffraction (XRD)

Diffraction peaks was seen at 31.64°, 34.31°, 36.12°, 47.43°, 56.48°, and 67.35°, per the results. This pattern clearly matches the phase of standard ZnO (JCPDS card No. 36-1451) [11], which is a hexagonal wurtzite polycrystalline structure with lattice planes (hkl) of (100), (002), (101), (102), (110), (103), and (112), as illustrated in Figure 1.

![Figure 1: XRD pattern of ZnO nanoparticles](image)

However, there were no peaks of other contaminants found. The observed ZnO spectra in various phases corroborated the high purity of the synthesised ZnO using the green synthesis approach [12]. A high crystallinity of ZnO was revealed by the presence of strong and narrow diffraction peaks in the XRD pattern [13]. The Debye–Scherer equation was used to compute the crystallite size of ZnO NPs, which was found to be 36.18 nm.

3.2 Scanning Electron Microscopy (SEM)

Figure 2 shows a SEM micrograph of the synthesised ZnO NPs. The formation of spherical nanoparticles with large agglomeration was observed in SEM images of ZnO NPs obtained using the green synthesis method. Kim and Park [14] and Ong et al. [15] found the exact structure in ZnO nanoparticles. During synthesis, the capping agent could be utilised to minimise particle size. The size of nanoparticles were determined using a scanning electron microscope. The particle size ranged from 37.77 to 64.13 nm, as indicated in Figure-2 by the SEM micrograph [16].
3.3 Energy Dispersive X-Ray Analysis (EDAX)

The proportional elemental composition of ZnO nanoparticles was confirmed using the energy dispersive X-rays Analysis (EDAX) tool, as illustrated in Figure 3, by measuring the intensity of the characteristic emitted x-rays. In synthesised zinc oxide nanoparticles, EDAX revealed only the presence of two elements: zinc and oxygen. The atomic and weight percent compositions of elements were shown in Table 1. The purity of the synthesised ZnO NPs was confirmed by EDAX analysis, and our findings are consistent with those of other studies Raj and Jayalakshmy et al. [17, 18].
Table 1: Elemental composition of ZnO nanoparticles

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>23.09</td>
<td>53.18</td>
</tr>
<tr>
<td>Zn</td>
<td>69.93</td>
<td>39.42</td>
</tr>
</tbody>
</table>

3.4 Fourier Transform Infrared spectroscopy (FTIR)

Synthesized zinc nanoparticles were subjected to FT-IR analysis to detect the various characteristic functional group associated with the synthesized nanoparticles. FTIR spectrum of the synthesized ZnO nanoparticles showed in Figure-4. The peaks indicate the characteristics functional group present in the synthesized zinc oxide nanoparticles. It is inferred that the samples have absorption peaks in the range of 3533.59 cm\(^{-1}\), 3321.42 cm\(^{-1}\), 1178.71 cm\(^{-1}\), 1045.42 cm\(^{-1}\), and 410.84 cm\(^{-1}\)[16]. The absorption peak at 410.84 to 575.9 cm\(^{-1}\) corresponds to metal-oxygen (ZnO stretching vibrations) vibration mode. The peak at 1045.42 cm\(^{-1}\) is ascribed to the stretching vibration of C-N bond of the primary amine or to the stretching vibration of the C-O bond of the primary alcohol. The peak at 1178.71 cm\(^{-1}\) was ascribed to primary alcohol in-plane bend or vibration. The peaks at 3321.42 cm\(^{-1}\) and 3533.59 cm\(^{-1}\) are ascribed to the stretching vibration of hydroxyl compounds [19, 20].

![Figure 4: FTIR spectra of ZnO nanoparticles](image)

4. Conclusion and Discussion:

ZnO nanoparticles were successfully synthesized by green synthesis method using Zinc nitrate hexahydrate and wheat grass pieces. SEM analyses revealed that the synthesized ZnO was spherical in shape and particle size ranged from 37.77 to 64.13 nm. The crystallite size and purity of the sample are investigated by XRD. In FTIR spectroscopy, pure ZnO nanoparticles showed stretching vibrations and peaks were match with standard fictional groups of ZnO.
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References:


