Green Synthesis and Characterization of Zinc Oxide Nanoparticles using Corchorus olitorius Leaf Extract

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Abstract – Green synthesis of metal oxide nanoparticles has gained prominence in recent years, resulting from the absence of toxic chemicals, low energy requirement, and eco-friendliness. This paper reports the green synthesis of zinc oxide nanoparticles (ZnO-NPs) using plant extract as a reducing agent. The ZnO-NPs were synthesized using Corchorus olitorius leaf extract and zinc acetate dihydrate, Zn (CH₃COO)₂·2H₂O as precursor. The synthesized ZnO-NPs were characterized by the application of UV–Vis spectroscopy, Transmission Electron Microscopy (TEM), X-ray diffraction (XRD), Energy Dispersive X-ray Spectroscopy (EDX) and Fourier Transform Infrared Spectroscopy (FTIR). UV–vis indicated the reduction of zinc acetate dihydrate into ZnO-NPs by the leaf extract. XRD and TEM revealed that the average size of the synthesized ZnO-NPs was 22 nm. The XRD pattern showed the hexagonal wurtzite crystalline nature of the synthesized ZnO-NPs. The elemental composition obtained from EDX showed that the synthesized ZnO-NPs are primarily composed of three elements: Zn (75.20%), O (20.48.7%), and C (4.32). Examination of stretching and bonding in the ZnO-NPs using FT-IR revealed the presence of Zn-O bonding at 430.37–403.93 cm⁻¹.

Keywords: Green synthesis; Zinc oxide; Nanoparticles; Characterization; crystalline; metal oxide.

Introduction

The prefix ‘nano’ is a Greek prefix meaning ‘dwarf’ or something very small and depicts one thousand millionth of a meter. Nanoscience is the study of structures and molecules on the scales of nanometers ranging between 1 and 100 nm, and the technology that utilizes it in practical applications is known as nanotechnology (Mansoori and Fauzi, 2005). Most scientists have come to believe that nanomaterials are one of the mainstays of emerging science and technology (Zhu et al., 2019; Wu et al., 2019). Due to larger specific areas, low modification temperature, tunable pore size, shorter distance of interparticle diffusion, and large number of associated adsorption sites compared to other materials, nanoparticles are considered to be remarkable absorbents and catalysts that can be used in environmental remediation (Gong et al., 2018). The narrow bandgap of nanoparticles can enhance their application as photocatalysts for degrading toxic organic contaminants and dyes (Eddy et al., 2022 and 2023). Other applications include biomedical applications (Mirzaei and Darroudi, 2017) and the industrial revolution, especially the food industry (Khan et al., 2019).

Zinc oxide is of great economic and industrial interest due to a wide range of properties that allows its application in many different areas, such as the rubber industry, environmental remediation, the biomedical field, and metal surface treatment (Gujel et al., 2017 Kathalewar et al., 2013, Pasquet et al., 2014, Xie et al., 2011, Zhang et al., 2013). Among many attributes of zinc oxide, the main characteristics are its semiconductivity, antimicrobial activity, vulcanization activation, and UV absorption (Pasquet et al., 2014; Koteswara, 2015; Kumar and Lee et al., 2016; Xie et al., 2018; Zaki et al., 2018; Zhang et al., 2018; Ahmoum et al., 2019; Grasland et al., 2019). With the introduction of nanotechnology, it is possible to enhance these properties once the surface area of the
material increases with the reduction in particle size and by manipulating the morphology of the nanomaterial (Kumar and Rani, 2013; Goh et al., 2014; Bandeira et al., 2020).

In general, nanoparticles are synthesized by physical and chemical methods. In the physical method, physical forces attract nano-scale particles and form large, stable, well-defined nanostructures. Its example includes nanoparticle synthesis through the colloidal dispersion method. It also includes basic techniques like vapor condensation, amorphous crystallization, physical fragmentation, and many others (Agarwal et al., 2017). Physical methods employ much energy, which is less economical (Iravani et al., 2011). The chemical method involves using toxic chemicals, which can be hazardous to the environment and humans involved in its handling. The literature has found that some of the toxic chemicals used in nanoparticle synthesis are hazardous in nature, causing several environmental issues. Therefore, the biological approach of nanoparticle formation has emerged as an excellent alternative synthesis method, which includes the application of plant products and their isolates, extracts, and different microbes. Given that this method is cost-effective, eco-friendly, and requires less energy, it is often classified as the most suitable for the synthesis of nanoparticles (Saravanan et al., 2018).

The interest in synthesizing nanoparticles via biological methods has increased considerably in the last decade. The development of this new approach and its significant interest are mainly related to the absence of toxic chemicals and less energy required, making the process more cost-effective and eco-friendly (Khalid et al., 2017; Krol et al., 2017). Consequently, this biological method of synthesis has become known as green synthesis. The term ‘green synthesis’ is associated with biological synthesis because this method follows the principles of green chemistry. The main advantages of green synthesis are the engagement of renewable sources, safer solvents, and auxiliaries while producing safer chemicals. Essentially, green synthesis uses biological substrates such as plants, bacteria, fungi, and algae to replace chemical solvents and stabilizers to decrease the toxicity of both product and process (Krol et al., 2017).

Recently, the use of plants for the green synthesis of nanomaterials has been explored extensively. In this method, various parts of the plants, such as the root, stem, leaf, seed, fruit, peel, and flowers, and their extracts are used to synthesize nanoparticles (Huynh et al., 2020). Plant constituents, including protein, amino acids, organic acid, polysaccharides, and secondary metabolites such as polyphenols, flavonoids, alkaloids, heterocyclic, and terpenoid compounds, are reducing and stabilizing agents. Despite the eco-friendly advantage of synthesizing nanoparticles from microorganisms, the following are their limitations: toxicity of some bacteria, difficulties in isolation, and incubation process (Mali et al., 2019). Therefore, plants remain the ideal potential for metal and metal oxide nanoparticle synthesis; this plausibility is attributed to rapid reaction rate with low energy, the occurrence of several biomolecules, cost-effectiveness, good stability, absence of hazardous chemicals, safe and easy operation procedures (Akintelu et al., 2020). This research, therefore, aimed at synthesizing zinc oxide nanoparticles (ZnO-NPs) via the green synthesis method using Corchorus olitorius leaves extract as the reducing agent and characterization of the synthesized ZnO-NPs.

Materials and Methods

Materials

Corchorus olitorius plant leaves were collected in a new clean polyethylene bag from a Samaru, Zaria, Nigeria garden. The leaves were transported to the laboratory immediately for the green synthesis process. Zinc acetate dihydrate, Zn (CH3COO)2.2H2O used was of analytical grade from Sigma-Aldrich Chemicals. Deionized water was used throughout the experiment.

Preparation of the Plant Extract

Sample preparation was done using the procedure reported by Meva et al. (2016) and Jayachandran et al. (2021) with little modification in terms of heating temperature of 70 °C instead of 80°C and centrifuge speed of 2200 rpm instead of 2400 rpm. The Corchorus olitorius leaves were washed thoroughly with distilled water 3 times to remove dust and other materials and air dried in the shade for two weeks. After drying, 10.00 grams of dried leaves were ground into powder form, mixed with 200 ml of distilled water, and heated at a temperature of 70 °C for 5 minutes to get the plant extract. The extract was cooled for 4 hours at atmospheric temperature and filtered using Whatman No. 1 filter paper. The aqueous extract was then
centrifuged at 2200 rpm for 5 min. The supernatant was stored at 4 °C for green synthesis of the ZnO-NPs.

**Green synthesis of zinc oxide nanoparticles (ZnO-NPs)**

To synthesize ZnO-NPs, 20 cm$^3$ of a zinc acetate dihydrate stock solution, Zn (CH$_3$COO)$_2$.2H$_2$O (0.1 M) was dissolved in 50 cm$^3$ deionized water. The mixture was stirred without heating for 30 minutes on a magnetic stirrer to dissolve the zinc acetate salt completely. After that, 20 cm$^3$ of aqueous leaf extract was added in drops to the zinc acetate solution and kept on a magnetic stirrer. After the addition of leaf extract, the mixture was heated at 60°C (Ekwumembo et al., 2023) and stirred for 4 hours. After this, the mixture was heated on a hot plate until it turned into yellowish-brown jelly, suggesting the presence of ZnO-NPs (Sujatha et al., 2018; Jayachandran et al., 2021). The jelly product was dried completely and subjected to calcination at 400 ◦C for 3 h. After calcination, The ZnO-NPs were obtained in a white-colored powder form, which was used for further characterization for confirmation of ZnO-NPs by UV–Visible spectroscopy (UV-vis), Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), X-Ray diffraction (XRD) and Energy Dispersive X-ray Spectroscopy EDX.

**Results**

**UV–Visible Spectroscopy of the Green Synthesized ZnO-NPs**

Figure 1 represents the UV–visible absorption spectrum of the green synthesized ZnO-NPs from Corchorus olitorius. UV–visible spectral analysis was performed with an Agilent Cary 300 U.V spectrophotometer, a single beam diode array spectrometer that collects spectra in the range 200–1100 nm using a slit width of 1 nm. The absorbance peak centered around 265 nm; this reveals that zinc acetate dehydrate has been reduced to zinc oxide nanoparticles (ZnO-NPs). The addition of Corchorus olitorius leaves extract solution to zinc acetate dihydrate [Zn (CH$_3$COO)$_2$.2H$_2$O] resulted in color change (yellowish brown) due to the formation of ZnO-NPs (Jayachandran et al., 2021). The color change arises from the excitation of surface plasmon vibrations with the zinc oxide nanoparticles (El-Belely et al., 2021).

![Figure 1. UV-vis spectra of the synthesized ZnO-NPs.](image)

**Transmission Electron Microscopy (TEM) of the Green Synthesized ZnO-NP**

Transmission electron microscopy (TEM) is essential in determining nanoparticle size, shape, and aggregation. The TEM images of the green synthesized ZnO-NPs (at different magnifications) are shown in Figure 2. The TEM image shows that the synthesized ZnO-NPs were in various shapes with faceted surfaces.
Figure 2. TEM micrograph of the synthesized ZnO-NPs at different magnifications.

X-ray diffraction (XRD) analysis of the synthesized ZnO-NPs

Figure 3 presents the XRD pattern of the green synthesized ZnO-NPs. XRD is one of the most important characterization techniques used in revealing the structural properties and size of NPs. When electrons have adequate energy for the dislocation of inner shell electrons of target materials, characteristic X-ray spectra are created because every crystalline substance has a characteristic atomic structure (Reddy et al., 2017; Vaishnav et al., 2017). XRD provides information on average particle size (D) through the Debye Scherrer formula given as 

$$D = \frac{0.9 \lambda}{\beta \cos \theta}$$

where \(\lambda\) = wavelength of X-ray, \(\beta\) = Full width at half maximum (in radians), \(\theta\) = Bragg’s diffraction angle (Khan et al., 2017a; Khan et al., 2017b; Ullah et al., 2017; Vaishnav et al., 2017).

Figure 3. XRD patterns of the green synthesized ZnO-NPs.
Energy-dispersive X-ray Spectroscopy (EDX)

The EDX spectra revealed the quantitative and qualitative elemental composition of the synthesized ZnO-NPs. Figure 4 shows the EDX spectra of the ZnO-NPs; the EDX result reveals that the synthesized ZnO-NPs are composed of three elements which are zinc, Zn (75.20%), Oxygen, O (20.48.7%), and carbon, C (4.32%).

Figure 4. EDX spectra of the synthesized ZnO-NPs

Fourier Transform Infrared Spectroscopy (FTIR)

Figure 5 shows the FTIR spectra of the green synthesized ZnO-NPs in the range 400 – 4000 cm⁻¹. The spectra reveal functional groups of the compounds on the nanoparticle's surface and in the synthesis solution and indicate the presence of ZnO nanoparticles.

Figure 5. FTIR Spectral of the green synthesized ZnO-NPs

Discussion

Provide a concise and precise description of the experimental results, their interpretation, and the experimental conclusions that can be drawn. Each Figure and Table should be supplied within the article itself. All Figures (charts, diagrams, and line drawings) and Images (photographic images) should be precise, in black and white, and numbered consecutively with Arabic numerals. Figures created in MS Word, MS PowerPoint, MS Excel, Illustrator, and Freehand should be saved in native formats.

Tables should be typed and included as part of the manuscript. They should not be submitted as graphic elements. Supply succinct and clear captions for all tables, figures, and plates. Ensure that any
superscripts or asterisks are shown next to the relevant items and have corresponding explanations displayed as footnotes to the table, figure, or plate. An example of the figure in the text is shown in Figure 2.

![Figure 2](image)

**Figure 2.** A pattern of observational rainfall and model: (a) uncorrected daily mean, (b) corrected daily mean, (c) uncorrected monthly mean, (d) corrected monthly mean.

An example of a table is shown in Table 1.

**Table 1.** Comparison of Statistical Analysis of the mean rainfall observations and models (Uncorrected and Corrected).

<table>
<thead>
<tr>
<th>Model</th>
<th>Uncorrected</th>
<th>Corrected</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>R²</td>
<td>NSE</td>
</tr>
<tr>
<td><strong>Mean daily rainfall</strong></td>
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<td></td>
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<td>ACCESS1-0</td>
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<tr>
<td>bcc-csm1-1</td>
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<tr>
<td>BNU-ESM</td>
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<tr>
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</tr>
<tr>
<td>CCSM4</td>
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<td>-3.99</td>
</tr>
<tr>
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</tr>
<tr>
<td>CSIRO-Mk3-6-0</td>
<td>0.46</td>
<td>-44.67</td>
</tr>
</tbody>
</table>
UV–Visible Spectroscopy of the Green ZnO-NPs

From the UV–UV-visible spectroscopy result, as presented in Figure 1, it was observed that the absorbance peak centered around 265 nm, indicating the reduction of zinc acetate dihydrate into ZnO-NPs. The absorbance peak observed in this work is higher than the 212 nm observed by Chinnasamy (2018)] and agrees with the 260 nm observed by Baesssa (2016). However, the absorbance peak of 265 nm observed is lower than the 370 nm and 382 nm observed by El-Belely et al. (2021) and Singh et al. (2019), respectively. This could have been caused by the percentage weight content of the ZnO-NPs in the sample.

Transmission Electron Microscopy of the ZnO-NP

The TEM image in Figure 2 shows that the synthesized ZnO-NPs were in various shapes with faceted surfaces, indicating high crystallinity. Some shapes that could be identified from the TEM image were spherical, cubic, and hexagonal. The size distribution was obtained by measuring the size of 100 randomly picked nanoparticles utilizing the ImageJ software. It was found that the green synthesized ZnO-NPs had an average size of 22 nm. The average size is within the range (10 – 30 nm) obtained by Singh et al. (2019) and is below the range (30 – 50 nm) obtained by El-Belely et al. (2021). The disparity in size could have resulted from the interplay between the different biomass and precursor used by other researchers as the reducing capability of different biomass used in green synthesis differs.

X-ray diffraction (XRD) analysis

Based on the XRD pattern presented in Figure 3, the green synthesized ZnO-NPs have a high purity of hexagonal crystalline structure as the diffraction peak is intense and narrower. Figure 4 shows the X-ray diffraction (XRD) pattern of the synthesized ZnO-NPs. The intensity peaks of the XRD pattern matched with those recorded in the Joint Committee on Powder Diffraction Standards (JCPDS, card No. 96-210- 7060), and it was confirmed that the synthesized nanoparticles are ZnO as the diffraction peaks of the nanoparticles tally with the characteristic hexagonal wurtzite structure of zinc oxide nanoparticles. The XRD pattern of synthesized nanoparticle was measured in the 2θ range (10-90°) as presented in Figure 3, an expected theta range to examine the crystalline structure of ZnO-NPs. The diffraction peaks of the ZnO-NPs indexed as 32.11°, 33.73°, 34.69°, 56.79°, and 59.89° correspond to (100), (002), (101), (110), (201) planes of hexagonal wurtzite crystalline nature with a preferred (101) orientation which has been observed by other authors (El-Belely et al., 2021). From the Debye Scherer formula, the average particle size obtained was 22 nm; this corresponds with what was obtained from TEM analysis in this study and also agrees with the 22 nm average nanoparticle size obtained by Vaishnav et al. (2017) when green synthesis of zinc oxide nanoparticles by Celosia argentea and its characterization was studied.

Energy-dispersive X-ray Spectroscopy (EDX) of Green synthesized ZnO-NPs.

This result has confirmed that the Zn-O-NPs synthesized have high purity. A similar finding was also reported in previous studies by Shamhari et al. (2018), where Zn (76.3%) and O (23.7%) were observed, and by Brintha and Ajitha (2015), where the mass percentage of Zn (73.9%) and O (26.1 %) was obtained. The other element (carbon) served as a capping organic agents bound to the surface of the zinc oxide nanoparticles. The high amount of Zn indicates that the synthesized nanoparticle is a good source of Zn for plants through absorption from soil during growth (Okon et al., 2023).

Fourier Transform Infrared Spectroscopy (FTIR) of the Green synthesized ZnO-NPs.

The Fourier transform infrared spectroscopy (FTIR) shows typical bands at 3300–3400 correspond to O-H stretching of alcohols, 3017.46 corresponds to =C-H stretching of alkenes, and C-H stretching of aromatic compounds as it falls within 3100 – 3000. 2775.81 corresponds to C–H stretching of aldehyde, 1982.37 indicates C-H bending of aromatic compounds, 1586.3 represents N-H bending of amine, 1264.83 is a muscular C-O stretching of ether group, 1016.07 falls within 1300 – 1000 corresponding to C–O stretching of esters and carboxylic functional groups (Jayachandran et al., 2021). The multiple peaks at 430.37 cm⁻¹, 423.49 cm⁻¹ and 403.93 cm⁻¹ correspond to Zn-O stretching. This aligns with the 414.87 cm⁻¹ observed for ZnO by Shamhari et al.
al. (2018) when the synthesis and characterization of zinc oxide nanoparticles with small particle size distribution was carried out.

**Conclusion**

The study reports green synthesis and characterization of zinc oxide nanoparticles (ZnO-NPs) from *Corchorus olitorius* leaf extract and zinc acetate dihydrate, Zn(CH$_3$COO)$_2$·2H$_2$O as the precursor. The synthesized ZnO-NPs were characterized by the application of UV–Vis spectroscopy, Transmission Electron Microscopy (TEM), X-ray diffraction (XRD), Energy Dispersive X-ray Spectroscopy (EDX) and Fourier Transform Infrared Spectroscopy (FTIR). UV-vis indicated the reduction of zinc acetate dihydrate into ZnO-NPs by the *Corchorus olitorius* leaf extract. XRD and TEM revealed that the average size of the synthesized ZnO-NPs is 22 nm. The XRD pattern showed the hexagonal wurtzite crystalline nature of the synthesized ZnO-NPs. The elemental composition obtained from EDX showed that the synthesized ZnO-NPs are primarily composed of three elements: Zn (75.20%), O (20.48.7%), and C (4.32). Examination of stretching and bonding in the ZnO-NPs using FT-IR revealed Zn-O bonding at 430.37–403.93 cm$^{-1}$

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**References**


