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Eco-friendly Synthesis of Zinc Oxide Nanoparticles Using *Camellia sinensis* (Green Tea) and Its Characterization

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Nanoparticles can be synthesized in a simple and eco-friendly way without any toxic byproducts through a process known as green synthesis. Green tea is a great source of polyphenols and flavonoids which can be used to synthesize ZnO nanoparticles. These nanoparticles have potential applications in various industries and medicine. Green tea's phenolic compounds act as efficient

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reducers of metal ions, which stabilizes the growth of nanoparticles. The study synthesized ZnO nanoparticles using green tea leaves and then characterized them using various instruments. The average particle diameter of the synthesized particles was 168.8 nm, and they had a zeta potential of 38.2 mV. The UV-Vis analysis showed a blue-shifted absorption maximum at 323 nm, indicating that the ZnO particles were on a nano-scale. The XRD spectra of calcined ZnO nanoparticles showed prominent diffraction peaks at various angles. Thermal stability of the ZnO nanoparticles was confirmed by the thermogravimetric analysis, which revealed that the nanoparticles degraded rapidly at 657°C, and weight loss began at 753°C.

Keywords: Zinc oxide; polyphenols; nanoparticles; TGA; TEM.

1. INTRODUCTION

Materials that are smaller than 100 nm are known as nanomaterials, and they exhibit atom-like behaviours due to their large surface area and wider band gap. Transition metal oxides and semiconductors that fall within the nanometer range have piqued interest in various fields [1]. Green synthesis is a method that reduces pollution and also offers several benefits, including simplicity, low cost, stable nanoparticles, quick production time, non-toxic byproducts, and the ability to synthesize on a large scale. There is an increasing demand for eco-friendly methods that do not involve the use of toxic materials in the synthesis process.

Green tea is abundant in polyphenols, particularly catechins, and other flavonoids. It is prepared without fermentation to prevent oxidation of these compounds. The composition of green tea is similar to that of a fresh leaf, with some changes occurs during drying. Green tea contains phenolic compounds, proteins, amino acids, carbohydrates, lipids, and vitamins C and E [2]. Phenolic compounds possess significant antioxidant potential and act as effective reducers of metal ions, making them ideal for supporting the eco-friendly synthesis of nanoparticles. Furthermore, the high level of proteins, lipids, and amino acids contribute to stabilizing nanoparticle growth and preventing particle agglomeration. ZnO NPs are metal nanoparticles with impressive properties, such as wide band gap, high piezoelectric property, and large binding energy. They are safe, non-toxic, and biocompatible, making them useful in various industries, including optoelectronics, diagnosis, and environmental protection. ZnO NPs can have potential applications in medicine as anti-angiogenesis, anti-inflammatory, and anticancer agents. Their surface is rich in -OH groups, allowing them to dissolve in both acidic and strong basic conditions [3].

This study focused on synthesis of ZnO nanoparticles using green tea leaves and subsequently characterize them using Particle Size Analyzer (Dynamic light scattering), Ultraviolet visible spectroscopy, X-ray diffraction, Thermogravimetric analysis, and Transmission electron microscopy.

2. MATERIALS AND METHODS

The precursor Zinc sulphate heptahydrate with 99% purity and Molecular weight 287.56 g/mol was obtained from Himedia. Distilled water was used throughout the experiment. Sodium Hydroxide (NaOH), was used as a stabilizing agent. Loose green tea leaves were obtained from Mighty Bio-Nature Products, Coimbatore.

2.1 Green Tea Extract Preparation

About 10 g of green tea leaves were weighed and made into fine powder. Then it was dissolved in 100 ml distilled water. It was boiled in a water bath at 65^0C for 3 hours. Then it was filtered using Whatman filter paper no.42 and then it was centrifuged at 5000 rpm for 15 min. Then this filtrate was stored at 5-10^{o}c for further experiments [4].

2.2 Synthesis of ZnO NPs

About 0.5 M Zinc sulphate solution was prepared by dissolving 14.38 g of $ZnSO_4$ in 100 ml of distilled water. To this 60 ml of green tea leaf extract was added dropwise until the colour changes from brownish to yellowish colour. It was followed by addition of 0.1 N NaOH dropwise which acts as a stabilizing agent. The precipitate was centrifuged at 8000 rpm for 15 minutes followed by two times washing with ethanol. Then oven dried at 90^0C for 12 hrs. The dried pellets were calcinated in a muffle furnace at 600^0C for 4 hrs to obtain white-coloured zinc oxide nanoparticles [1,3].

2.3 Characterization Techniques

The Synthesized ZnO NPs were characterized using PSA (Model HORIBA SZ-100), UV-Vis (Analytik Jena - Specord 210 Plus), XRD (The Empyrean Series III Diffractometer and TEM (Tecnai 12).

3. RESULTS AND DISCUSSION

The synthesized ZnO NPs were white in colour (Plate 1). The White colour of the nanoparticles is due to biomolecules and polyphenols present in the green tea leaf extract, which covers the surface of the nanoparticles [5].

Plate 1. Zinc Oxide Nanoparticles

3.1 Dynamic Light Scattering and Zeta Potential Analysis

Dynamic light scattering, also known as Photon correlation spectroscopy, determines particle size and particle size distribution by analyzing the diffusion coefficients of particles in a scattering volume and measuring the timescale of fluctuations in scattered light intensity. For ultra-low concentration systems, particle diffusion in and out of the volume can result in fluctuations in both Brownian motion and local particle concentration [6]. The Fig. 1 displays the particle size distribution, which was obtained through DLS analysis using the Origin software. The particle size and zeta potential was measured by PSA. The results showed the particle has average particle diameter of 168.8 nm in an aqueous colloidal solution. Additionally, the zeta potential of this particle was 38.2 mV. The result was coincided with result obtained by [7,8].

3.2 UV – Visible Spectrum Analysis

UV-Vis spectroscopy studies matter's interaction with electromagnetic radiation in the UV-visible region. It covers 10-380 nm and has three sub-regions: UVA (320-380 nm), UVB (280-320 nm), and UVC (100-280 nm). It uses electronic spectroscopy to excite outermost electrons and can interact with matter through absorption, reflection, and photoluminescence [9]. The UV-Vis spectra of ZnO NPs were analysed. It is depicted in Fig. 2. The significantly blue-shifted absorption maximum at 323 nm confirms the synthesis of ZnO product on a nano-scale. Typically, for bulk ZnO, the absorption maximum occurs around 380-385 nm. This result was as same as that of the results obtained by [10,11]. They explained that the reduction in the size of ZnO causes a decrease between two valence bands, thereby causing an increase in frequency which leads to a decrease in wavelength towards the blue end of the spectrum.

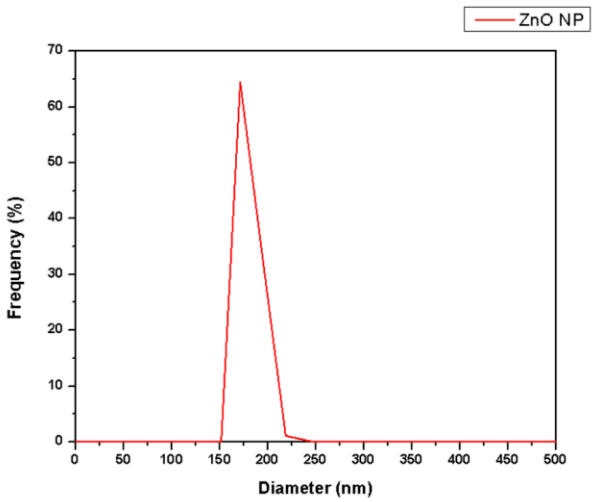

Fig. 1. Particle size analysis of biosynthesized zinc oxide nanoparticles

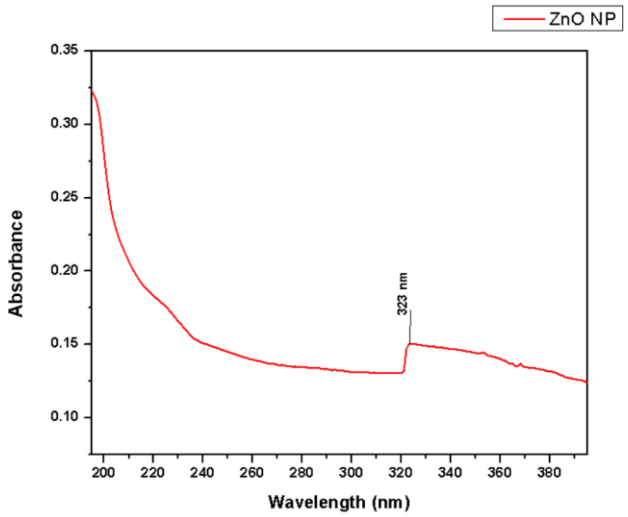

Fig. 2. Ultraviolet absorption spectrum of biosynthesized zinc oxide nanoparticles

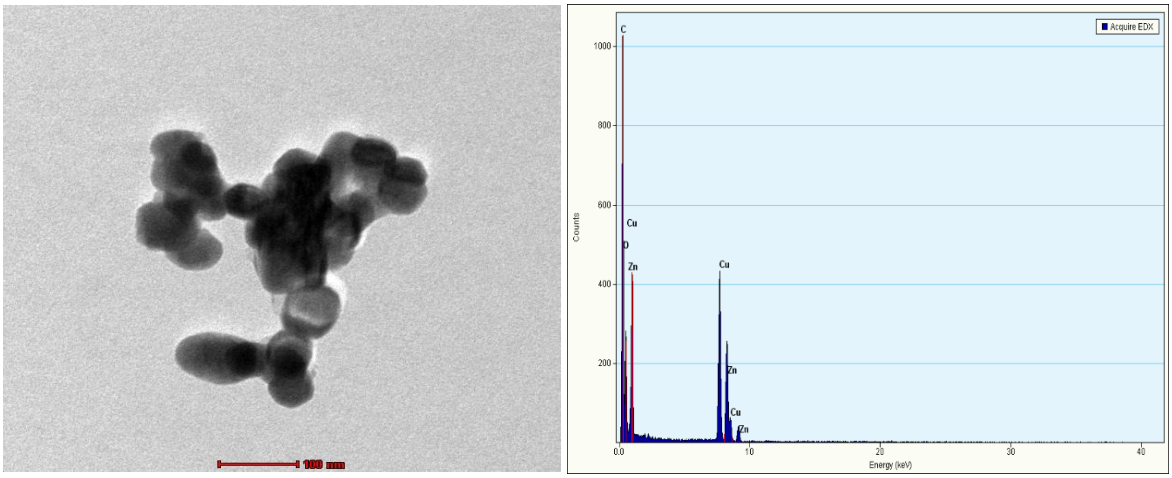

Fig. 3. TEM image & EDAX image of biosynthesized zinc oxide nanoparticles

3.3 TEM Analysis

The morphology of ZnO nanoparticles synthesized was observed using TEM micrograph. The TEM micrograph of the ZnO NPs confirmed that the particles were almost spherical in shape at the resolution of 100 nm. Energy dispersive X-ray analysis confirmed the presence of zinc and oxygen groups in the sample [12,13].

3.4 XRD Analysis

Powder X-ray diffraction (XRD) is the most widely used and essential technique for characterizing nanoscale materials. It provides crucial information that is unmatched by other microscopic and spectroscopic methods, including accurate phase identification, precise sample purity, crystallite size, and morphology analysis, among others [14]. The XRD analysis was done for ZnO NPs. The XRD spectra is given in Fig. 5. The ZnO NPs were calcinated before getting XRD spectra. The diffraction peaks that are most prominent are (100), (002), (101), (102), (110), (103), and (112) with diffraction angles of 31.78^0, 34.34^0, 36.18^0, 47.63^0, 56.53^0, 62.79^0, and 67.92^0, respectively. The average particle size (D) of the synthesized nanoparticles was calculated using the Scherrer formula, which is well known. These XRD results were compatible with [15,16,17] that confirms the prepared ZnO sample is highly crystalline with similar predominant peaks at diffraction angles of 31.78^0, 34.34^0 & 36.18^0.

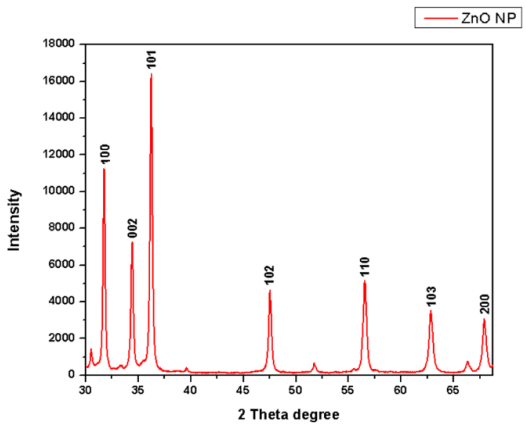

Fig. 4. XRD analysis of biosynthesized zinc oxide nanoparticles

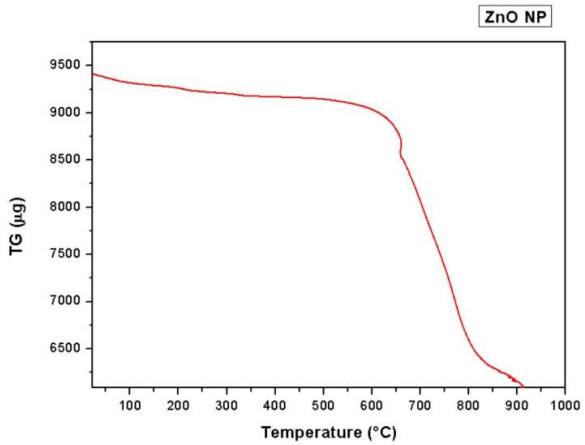

Fig. 5. Thermogravimetric analysis of biosynthesized zinc oxide nanoparticles

3.5 Thermogravimetric Analysis (TGA)

To determine the effects of high temperature on a specific sample, thermogravimetric analysis is utilized to measure its phase transition, absorption, adsorption, and desorption. In this study, ZnO nanoparticles were analyzed using a temperature program ranging from 27^0C to 900^0C and with a temperature interval of 20^0C per min. The TGA Curve of ZnO NPs is given in Fig. 5. The results indicated that at 657^0C, rapid degradation of the zinc oxide nanoparticles takes place, while at 753^0C, the material begins to experience weight loss. This indicates that the biosynthesized ZnO nanoparticles are thermally stable up to 600°C which is similar to [18,3].

4. CONCLUSION

The process of using *Camellia sinensis* as a reductant to synthesize ZnO NPs is impressive for its cost-effectiveness, speed, and eco-friendliness. The synthesized ZnO NPs had an average particle diameter of 168.8 nm in an aqueous colloidal solution, with a zeta potential of 38.2 mV. The blue-shifted absorption maximum at 323 nm in the UV-Vis spectrum confirms the particle in is nanoscale. The morphology of the ZnO nanoparticles was observed using TEM micrograph at a resolution of 100 nm, with energy-dispersive X-ray analysis confirming the particle is in spherical shaped and also presence of zinc and oxygen groups in the sample. Calcination at 600°C was done to attain higher crystallinity and to remove all water, as evidenced by the prominent diffraction angles observed at 31.78^0, 34.34^0, 36.18^0, 47.63^0, 56.53^0, 62.79^0, and 67.92^0 with miller indices (100), (002), (101), (102), (110), (103), and (112), respectively in the XRD spectra of the calcined ZnO NPs. The TGA graph of ZnO NPs revealing that rapid degradation of the zinc oxide nanoparticles occurred at 657°C and weight loss

began at 753°C indicating that ZnO nanoparticles are thermally stable upto 600^{0}C.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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