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Green Synthesis and Characterization of Copper Oxide Nanoparticles Using Tecoma Stans

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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

Aims: An eco-friendly approach utilizing biologically-mediated processes has been developed for the fabrication of CuO nanoparticles using *Tecoma stans* leaves.
Methodology: The synthesis of CuO nanoparticles was carried out using an environmentally friendly method, which involved the use of an aqueous solution of copper (II) sulfate and a leaf extract derived from *Tecoma stans*. The synthesized CuO nanoparticles were characterized using various instruments, including a UV-visible spectrophotometer, scanning electron microscopy, X-ray Diffraction Spectrometer, and Fourier-transform infrared spectroscopy.

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Results: The UV-Vis spectrum showed a characteristic absorption peak of CuO nanoparticles at 296 nm. The green-synthesized CuO nanoparticles were characterized using SEM, which revealed their morphological and structural characteristics and showed a mean particle size ranging from 20 nm to 200 nm. The FTIR spectrum, ranging from 584.63 cm-1 to 3430.41 cm-1, indicated the presence of various functional groups. X-ray powder diffraction (XRD) analysis confirmed the formation of monoclinic CuO with a crystalline nature, and an average particle size of 27.67 Å.
Conclusion: The present study reports the development of a simple, low-cost, and biologically-mediated method for synthesizing CuO nanoparticles using an extract obtained from *Tecoma stans* leaves. The effectiveness of these nanoparticles can be evaluated through various biological activities, including antibacterial, antifungal, and cytotoxic assays.

Keywords: Tecoma stans; green synthesis; nanoparticles; copper sulfate; characterization.

1. INTRODUCTION

Nanoparticles have garnered substantial significance in various domains of medical research, including biomedical research, biosensors, pharmaceuticals, catalysis, drug delivery, healthcare, cosmetics, household products, mechanics, optics, chemicals, and antimicrobial applications, owing to their superior physical, chemical, mechanical, thermal, and biological properties [1]. Metal nanoparticles can be nanosized metals, metal oxides, metal sulfides, or metal phosphates [2,3]. “Metal and metal oxide nanoparticles exhibit unique physicochemical properties that differ from their bulk counterparts, including distinctive surface, optical, thermal, and electrical properties. The synthesis of metal and metal oxide nanoparticles is achieved through the addition of reducing or oxidizing/precipitating agents” [4]. “Copper is an essential micronutrient found in various enzymes and proteins. Copper nanoparticles have diverse applications in commercial settings, such as antimicrobial agents, catalysts, gas sensors, electronics, batteries, and heat transfer fluids” [5].

Copper oxide possesses a monoclinic crystal structure and is classified as a p-type semiconductor due to its acceptor-type dopants. It has a narrow bandgap energy of 1.7 eV, which is responsible for its unique electronic properties and applications [6]. The synthesis of CuO nanoparticles can be achieved through various methods, including sol-gel, electrochemical, thermal decomposition, microwave irradiation, solid-state reactions, precipitation, solution combustion, ultrasonic mixing, and self-assembly techniques [7]. Copper and its complexes have a broad range of applications, such as water purification, algaecides, fungicides, and antibacterial agents. They are also widely used in the production of gas sensors, batteries, high-temperature superconductors, textile industries, thermosensing and conducting materials, catalysis, inorganic-organic nanocomposites, magnetoresistant materials, environmental remediation, and solar energy conversion devices [8]. “Green synthesis is recognized as a superior alternative to chemical or microbial synthesis due to its scalability and reduced complexity. It enables the efficient production of large quantities of desired nanoparticles without involving laborious and time-consuming processes” [9]. “Green synthesis of copper oxide nanoparticles using plants and plant extracts has garnered significant attention among researchers due to its numerous advantages over physical and chemical methods. This approach is characterized by its eco-friendliness, cost-effectiveness, and absence of toxic byproducts. Phytoconstituents present in plants serve as effective reducing and capping agents in this method” [10].

Tecoma stans, commonly known as yellow bell, is a member of the Bignoniaceae family and is distributed in tropical and subtropical regions worldwide. Extensive phytochemical investigations have been conducted on this plant, which has demonstrated the existence of numerous compounds, including alkaloids, iridoid glycosides, lapachol, and various primary and secondary metabolites such as sugars, triterpenoids, sterols, and phenolics [11,12]. The plant has various therapeutic properties, with different parts of the plant exhibiting different activities. The leaves, for instance, possess anthelmintic [13], antispasmodic [14], antibacterial [15], anticancer [16], and wound healing properties, while the flowers have antidiabetic [17], and anticancer activity [11]. The roots possess antibacterial activity, and the bark has wound-healing properties, while the aerial parts exhibit antioxidant activity.

To the best of our knowledge, there is currently no existing literature on the green synthesis of CuO nanoparticles utilizing *Tecoma stans* leaf extract. Therefore, the objective of this study is to examine the green synthesis of CuO nanoparticles utilizing *Tecoma stans* leaf extract and to conduct a comprehensive characterization of the nanoparticles using UV-Visible Spectroscopy, Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and X-ray diffraction analysis (XRD).

2. METHODS

2.1 Plant Collection and Authentication

The leaves of *T. stans* (commonly called yellow bells) were collected from the trees growing around the local areas of Salem, Tamil Nadu, India. The plant was identified and authorized by the Professor. Dr.K.Kannan, Taxonomist, Department of Botany, Vivekanandha College Of Arts And Sciences For Women, Namakkal, Tamil Nadu, India. The collected plant material was washed with double distilled water to eliminate any surface impurities and subsequently minced into small fragments. The leaves were then subjected to shade drying for 7-10 days.

2.2 Preparation of Plant Extract

The leaves were ground into a fine powder using a mixer grinder. “100 gm of the powder was then dispersed in 100 mL of distilled water and boiled at a temperature of 60ºC for 20 minutes. Following cooling, the extract was filtered using a Whatman No.1 filter paper and stored in a refrigerator for subsequent investigation” [18].

2.3 Green Synthesis of Copper Oxide Nanoparticles

Copper oxide nanoparticles were synthesized via a reaction between copper sulfate and plant extract. “A solution of 0.1 M copper sulfate in double distilled water was prepared. Copper sulfate and plant extract were then mixed in varying ratios of 5:5, 6:4, 7:3, 8:2, and 9:1. The resulting mixture was heated below boiling point and continuously stirred at 800 rpm using a magnetic stirrer. The mixture turned green in color within 1 hour. The reaction was conducted in darkness. The obtained suspension was centrifuged at 15,000 rpm for 15 minutes. The resulting pellet containing copper nanoparticles was washed with deionized water 3-4 times to eliminate impurities. The precipitated nanoparticles were lyophilized and stored in a cool, dry, and dark place for subsequent characterization” [19].

2.4 Characterization of Copper Oxide Nanoparticles

The copper oxide nanoparticles that were synthesized underwent several analytical techniques for characterization, which included Ultraviolet-Visible (UV-Vis) Spectroscopy, Fourier Transformed Infrared (FTIR) Spectroscopy, Scanning Electron Microscopy (SEM), and X-ray Diffraction analysis (XRD).

2.4.1 UV-visible spectral analysis

“The synthesized CuONPs were initially characterized by a UV-visible spectrophotometer to confirm their presence. Ultraviolet spectral measurement was carried out in the range of wavelength between 300-700 nm by double beam UV-visible spectrophotometer (PD-303 UV)” [20,21].

2.4.2 Fourier transform infrared spectral analysis

The FTIR spectra were acquired using a Shimadzu IR-Prestige 21 spectrophotometer to identify the bioactive molecules responsible for reducing copper ions and the capping ability of the bioreduced CuONPs. The FTIR spectra also enabled the determination of the functional groups present in the synthesized copper oxide NPs, as each chemical bond has an energy absorption band that can be used to investigate the structural and bonding information of the complex to determine the type and strength of bonding. The FTIR spectra of the synthesized samples were obtained using the KBr pellet method over a range of 4000-400 cm−1 with a resolution of 4 cm−1.

2.4.3 Scanning electron microscopy (SEM) analysis

The microstructure and particle size characteristics of CuONPs produced using the green approach were determined using field emission scanning electron microscopy (FESEM) with ZEISS Smart SEM equipment operating at 5.0 kV. A carbon ribbon was used to create a thin film onto which 1 mg of copper oxide nanoparticles was deposited and coated with

carbon. Images were captured at various magnifications to examine the microstructure and particle size characteristics. ImageJ software was employed to assess the nanoparticle size distribution based on the FESEM images.

2.4.4 X-Ray diffraction (XRD) analysis

X-ray diffraction measurements of CuO NPs were carried out using an X-ray diffractometer instrument [Shimadzu XRD-6000, AS (3k.NOPC)], an angle range between 30-80° with CuKα radiation in a θ-2θ configuration. The average crystallite size of the CuONPs is calculated by the Debye-Scherrer formula as, $D = k\lambda/\beta\cos\theta$, where D is particle diameter size, k is a constant equals 0.94, λ is the wavelength of X-ray source (0.1541 Å), β is the full width at half maximum (FWHM) and θ is the Bragg angle.

3. RESULTS AND DISCUSSION

3.1 UV-Visible Spectroscopy

The UV-Visible absorption spectrum of copper oxide nanoparticles synthesized through green methods using *Tecoma stans* is presented in Fig. 1. "The UV-visible spectrum of the synthesized CuO nanoparticle dispersed in water displays a robust absorbance of UV rays ranging from 200-300 nm, signifying the presence of CuO nanoparticles in the sample. The peak observed at 296 nm is attributed to surface plasmon absorption of CuO nanoparticles, which results from the combined oscillation of free conduction band electrons excited by incident UV radiation" [21]. The phenomenon of resonance occurs when the wavelength of the incident light is significantly larger than the diameter of the particles. The UV-visible spectra of CuO nanoparticles, produced by reacting copper sulfate solutions with leaf extract of *Tecoma stans*, are depicted in Fig. 2, with a peak observed at 809.05 nm. This peak suggests a distinct mechanism of particle excitation and absorption, possibly attributed to the use of plant extracts as reducing agents during the synthesis process [15].

3.2 FT-IR Analysis

The functional groups present in copper oxide nanoparticles (CuONPs) synthesized using green methods were identified through Fourier transform infrared (FTIR) spectroscopy. The FTIR spectrum of CuONPs synthesized from leaf extract of *Tecoma stans* was examined to determine the bioactive compounds responsible for capping and stabilizing the nanoparticles. The FTIR spectra, as shown in Fig. 3, exhibit bands at specific wavenumbers that correspond to various functional groups. The band at 584.63 cm-1 is attributed to the C-Cl stretch in an organic molecule containing a chloro substituent, while the peak at 932.69 cm-1 corresponds to the C-H and N-H bending vibration in aromatic compounds. The peak at 1019.64 cm-1 indicates the C-O stretching vibration in ethers, while the band at 1280.99 cm-1 corresponds to the C-N stretching vibration in amines. The peak observed at 1493.95 cm-1 represents the C=C stretching vibration in double bonds, and the peak at 1384.55 cm-1 is attributed to the C-H bending vibration in methyl and/or methylene groups. The peak at 1417.94 cm-1 represents the C-H bending vibration in methyl groups, while the band at 3430.41 cm-1 indicates the stretching vibration of the O-H bond in various organic molecules such as alcohols, phenols, and carboxylic acids. Finally, the peak at 2852.57 cm-1 shows the stretching vibration of C-H bonds in various organic molecules, such as alkanes, alkenes, and aromatic compounds.

3.3 Scanning Electron Microscope

The surface morphology of copper nanoparticles synthesized using *T. Stans* leaf extract was analyzed using Scanning Electron Microscopy (SEM). SEM analysis provides information about the morphology and size characteristics of the synthesized copper oxide nanoparticles. Fig. 4 shows the surface morphology of the CuO nanoparticles, which were found to be asymmetrically dispersed and occasionally aggregated into free crystal structures. Two SEM images were presented, one showing CuO NPs with a size of 2µm, and the other showing CuO NPs with a size of 200nm. The synthesized CuO nanoparticles exhibited a mixture of shapes, including spherical, hexagonal, and uneven shapes. The morphology of CuO nanoparticles can vary depending on their size, indicating a diverse range of shapes. However, the exact surface texture of the nanoparticles could not be determined. The prepared CuO nanoparticles displayed a nearly monodisperse distribution of particle sizes, with some evidence of lumped particles that formed bulky micron-sized aggregates.

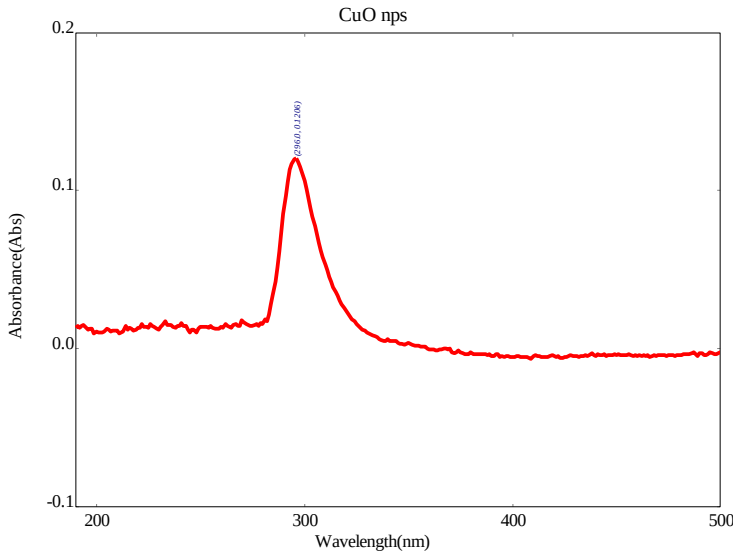

Fig. 1. UV-visible spectra of *Tecoma stans* mediated copper oxide nanoparticles

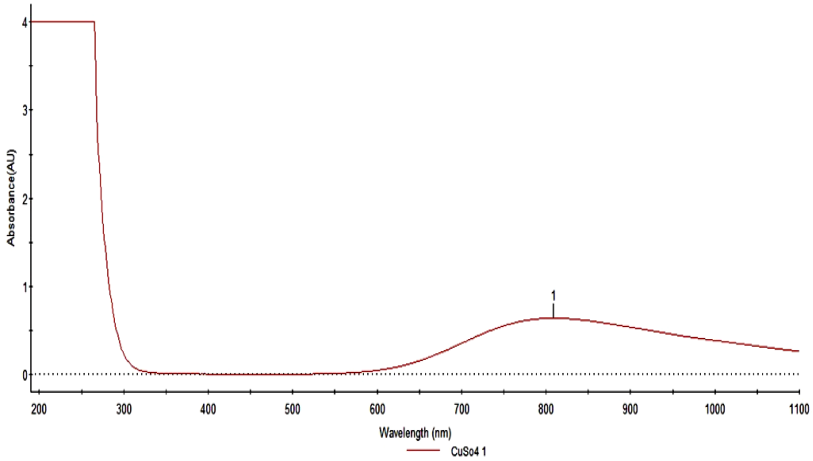

Fig. 2. UV-visible spectra of *the reaction mixture of leaf extract of Tecoma stans with copper sulfate* solutions

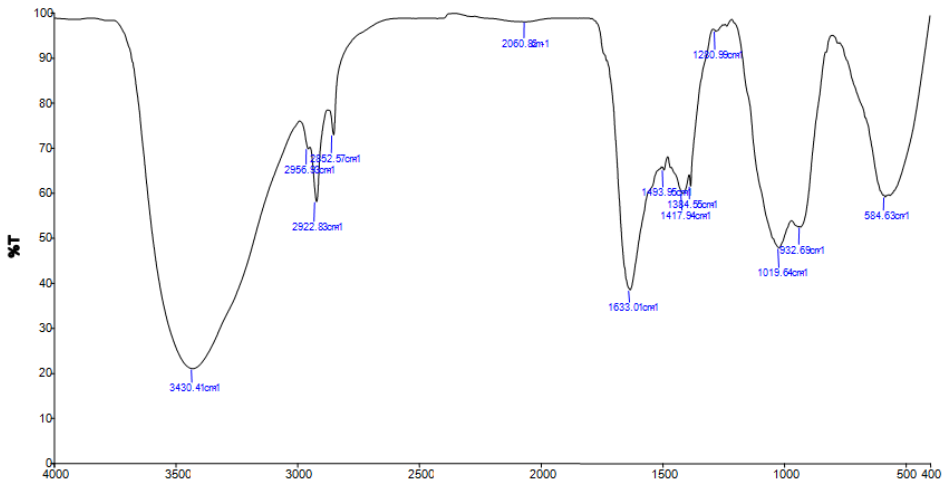

Fig. 3. FT-IR Analysis of *Tecoma stans* mediated copper oxide nanoparticles

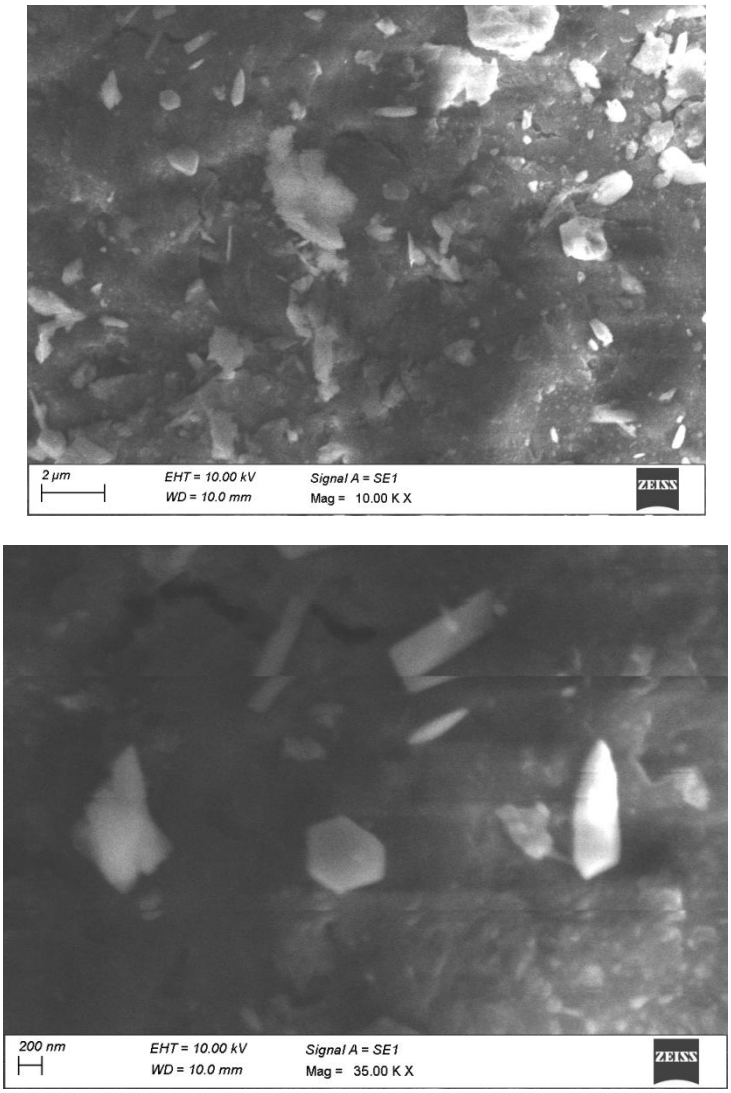

Fig. 4. SEM images of *Tecoma stans* mediated copper oxide nanoparticles

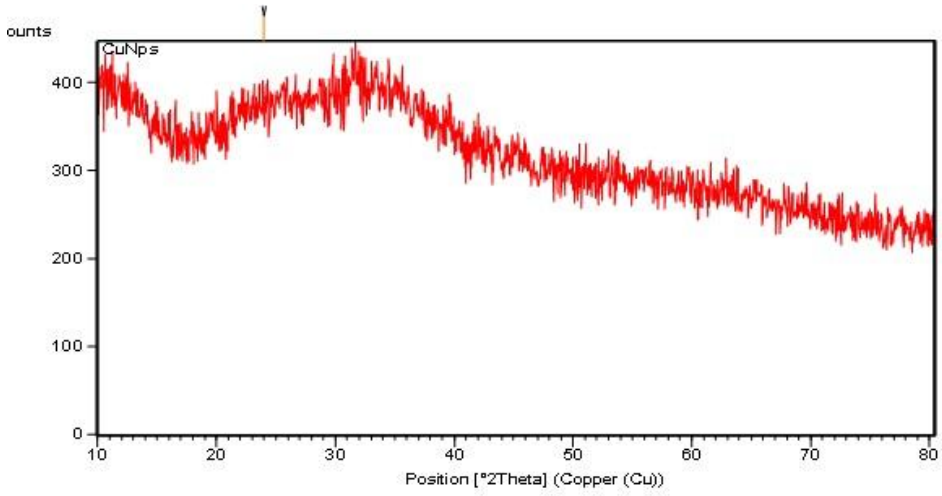

Fig. 5. X-Ray diffraction Analysis of *Tecoma stans* mediated copper oxide nanoparticles

3.4 X-ray Diffraction Analysis

Fig. 5 presents the results of the X-ray diffraction (XRD) analysis of CuO nanoparticles synthesized through green methods. The XRD spectra display two major peaks at 17°2θ and 32°2θ. The peak at 32°2θ corresponds to the (002) plane reflection of CuO nanoparticles and is a characteristic peak of CuO, indicating the formation of CuO nanoparticles. It provides information on the size and crystallinity of the particles based on the intensity and width of the peak. Likewise, the peak at 17°2θ also corresponds to the (002) plane of CuO crystal structure and indicates the crystalline nature of the nanoparticles. The Scherrer equation, which relates the average particle size to the X-ray powder diffraction data, can be used to calculate the average particle size. In this study, the XRD pattern showed an average particle size of 27.67 Å.

4. CONCLUSION

CuO nanoparticles were synthesized using *Tecoma stans* leaves through a green synthesis method. The synthesized nanoparticles were characterized using various analytical techniques, including UV-visible spectroscopy, FT-IR analysis, scanning electron microscope (SEM), and X-ray diffraction (XRD) analysis. The characterization results confirmed the successful synthesis of CuO nanoparticles and provided detailed information about their morphology, size, and crystallinity.

The UV-visible spectrum showed a strong absorbance of UV rays between the wavelength of 200-300 nm, which indicated the presence of CuO nanoparticles in the sample. The peak formed at 296 nm was due to the surface plasmon absorption of CuO nanoparticles. FT-IR analysis confirmed the presence of bioactive compounds that were responsible for the capping and efficient stabilization of CuO nanoparticles synthesized from leaves extract. SEM analysis showed that the synthesized copper oxide nanoparticles were dispersed asymmetrically and infrequently aggregated to form free crystal structures.

XRD analysis confirmed the formation of CuO nanoparticles and provided information about the size and crystallinity of the particles based on the intensity and width of the peak. The Scherrer equation was used to calculate the average particle size using X-ray powder diffraction data, which showed an average particle size of 27.67 Å.

Overall, the characterization results indicated that the synthesized CuO nanoparticles had a diverse range of shapes and sizes, which could vary based on their size. The presented analytical techniques can be used to analyze other nanoparticles synthesized using green methods and provide detailed information about their morphology, size, and crystallinity.

CONSENT

It is not applicable.

ETHICAL APPROVAL

It is not applicable.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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