Original Research Article

Green Synthesis and Characterization of Copper Oxide Nanoparticles using *Tecoma Stans* Plant Leaves

ABSTRACT

Aims: The objective of the study was to assess the characterization of copperoxide mediated nanoparticles of *Tecoma stans* leaves.

Methodology:

The bio-capping and reduction properties of *Tecoma stans* leaves extract were found to be highly effective in the bio-synthesis of copper oxide nanoparticles. The resulting biogenic copper oxide nanomaterials were analyzed and characterized using a range of techniques, including UV-Visible Spectroscopy, FT-IR, SEM, and XRD.

Results:

The characteristic absorption peak of CuONPs was in the range of 296 nm in the UV–Vis spectrum. SEM revealed the morphological and structural characteristics of the green-synthesized CuONPs with 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, confirms the presence of various functional groups. X-ray powder diffraction (XRD) analysis revealed the formation of monoclinic, crystalline nature of CuO with average particle of 27.67 Å

Conclusion:

In this study, a simple, biological, and low-cost method for the production of copper nanoparticles using *Tecoma stans* leaves extract was developed. To assess the effectiveness of these nanoparticles, they can be subjected to various biological activities, such as antibacterial, antifungal, and cytotoxic evaluations.

Keywords: Tecoma Stans, Green Synthesis, Nanoparticles, Copper Sulfate, Characterization

1. INTRODUCTION

Nanoparticles have gained significant medical importance in the fields of biomedical research, biosensors, pharmaceuticals, catalysis, drug delivery, healthcare, cosmetics, household products, mechanics, optics, chemicals, and antimicrobial applications due 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 possess distinct physicochemical properties from their native conformations, including 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 important micronutrient present in

enzymes and proteins. Copper nanoparticles are widely used as antimicrobial agents, catalysts, gas sensors, electronics, batteries, and heat transfer fluids commercially(5).

Copper oxide is characterized by a monoclinic structure and is classified as a p-type semiconductor with a narrow bandgap of 1.7 eV(6). The synthesis of CuO nanoparticles is achieved through sol–gel, electrochemical, thermal decomposition, microwave irradiation, solid-state reactions, precipitation, solution combustion methods, ultrasonic mixing, and self-assembly methods(7).Copper and Cu complexes are used as water purifiers, algaecides, fungicides, and antibacterial agents. They are widely used in gas sensors, batteries, high-temperature superconductors, textile industries, thermosensing and conducting materials, catalysis, the synthesis of inorganic-organic nanosize composites, magnetoresistant materials, high-temperature superconductors, environmental remediation, and solar energy conversion tools(8).Green synthesis is considered advantageous over chemical or microbial synthesis because it can be produced in large quantities and eliminates tedious processes(9). The green synthesis of copper oxide nanoparticles using plants and plant extracts is a major focus of researchers, and it is more advantageous than physical and chemical methods due to its clean, nontoxic, cost-effective, and environmentally friendly approach. The phytoconstituents present in the plants act as reducing and capping agents(10).

Tecoma stans, also known as yellow bell, is a plant belonging to the family Bignoniaceae and is found in tropical and subtropical regions worldwide. Various phytochemical studies have been conducted on this plant, revealing the presence of several compounds, including alkaloids, iridoid glycosides, lapachol, and other 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.

As far as we know, there are no reports available regarding the green synthesis of CuO-NPs utilizing the leaf extract of *Tecoma stans*. Therefore, this study aims to investigate the green synthesis of CuO-NPs using *Tecoma stans* leaf extract and to characterize the resulting 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, Vivekanadha College Of Arts And Sciences For Women, Namakkal, Tamil Nadu, India. The collected plant parts were rinsed with double distilled water to remove the dirt present on the surface and then chopped into small pieces. The leaves and flowers were allowed to shade dry for about 7-10 days.

2.2 Preparation of plant extract

The leaves were powdered using mixer grinder. The 100gm of powder disseminated in 100 ml of distilled water followed by boiling at 60ºC for 20 min. After cooled extract was filtered using Whatman No 1 filter paper and stored at refrigerator for further study(18).

2.3 Green Synthesis Of Copper Oxide Nanoparticles

Synthesis of copper oxide nanoparticles carried out using copper sulfate and Plant extract. 0.1 M copper sulphate in double distilled water. Copper sulfate and Plant extract were mixed together in a ratio of 5:5, 6:4, 7:3, 8:2 and 9:1. The reaction mixture was heated below the boiling point and continuously stirred at 800 rpm using magnetic stirrer. The mixture turned into green in color within 1 hr. The whole reaction was carried out in the dark. The obtained suspension was centrifuged at 15,000 rpm for 15 min. The pellet containing copper nanoparticles was washed 3–4 times with deionized water to remove impurities. The precipitated nanoparticles were lyophilized nanoparticles were stored in a cool, dry, and dark place and further their characterization was carried out(19).

2.4 Characterization Of Copper Oxide Nanoparticles

The synthesized copper oxide nanoparticles were characterized by various analytical methods includes Ultraviolet-Visible (UV-Vis) Spectroscopy, Fourier Transformed Infrared (FTIR) Spectroscopy, Scanning Electron Microscopy (SEM), X-ray Diffraction analysis(XRD).

2.4.1 UV-Visible Spectral Analysis

The synthesized CuONPs were initially characterized by UV-visible spectrophotometer to confirm its presence. Ultra violet spectral measurement was carried out in range of wavelength between 300-700 nm by double beam UV-visible spectrophotometer (PD-303 UV)(20).

2.4.2 Fourier Transform Infrared Spectral Analysis

The FTIR spectra was detected (Shimadzu IR-Prestige 21) to identify the bioactive molecules responsible for the reduction of copper ions with capping ability of the bioreduced CuONPs**.** It also helps to find out the functional groups present in synthesized copper oxide NPs, because each chemical bond has an energy absorption band used to examine the structural and bond information of complex to study bonding type and their strength. The FTIR spectra of synthesized samples were obtained by using the KBr pellet method, in the range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1}.

2.4.3 Scanning Electron Microscopy (SEM) Analysis

The Carl Zeiss Sigma equipment was used for field emission scanning electron microscopy (FESEM) at 5.0 kV to determine the microstructure, and particle size characteristics of the CuONPs produced using the green approach. A carbon ribbon was taken, and 1 mg of copper nanoparticles was laid as a thin film coated using carbon, and several magnifications were used to capture the images. ImageJ software was employed to assess nanoparticle size distribution based on FESEM images.

2.4.4 X-Ray Diffraction (XRD) Analysis

X-ray diffraction measurements of CuO NPs was carried out using 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 calculated by 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 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 *Tecoma stans* mediated copper oxide nanoparticles synthesized through green methods is depicted in Fig. 1. The UV-visible spectrum of the synthesized CuO nanoparticle dispersed in water shows strong absorbance of UV rays between 200-300 nm, indicating the presence of CuO nanoparticles in the sample. The peak formed at 296 nm is due to surface plasmon absorption of CuO nanoparticles, which is caused by the combined oscillation of free conduction band electrons excited by incident UV radiation(21). This resonance occurs when the wavelength of the incident light is much larger than the particle diameter.

The UV-visible spectra of CuO nanoparticles, synthesized from the reaction mixture of copper sulfate solutions with leaf extract of *Tecoma stans*, are illustrated in Fig. 2, with a peak observed at 809.05 nm. This peak indicates a different mechanism of particle excitation and absorption, possibly due to the use of plant extracts as reducing agents during synthesis (15).

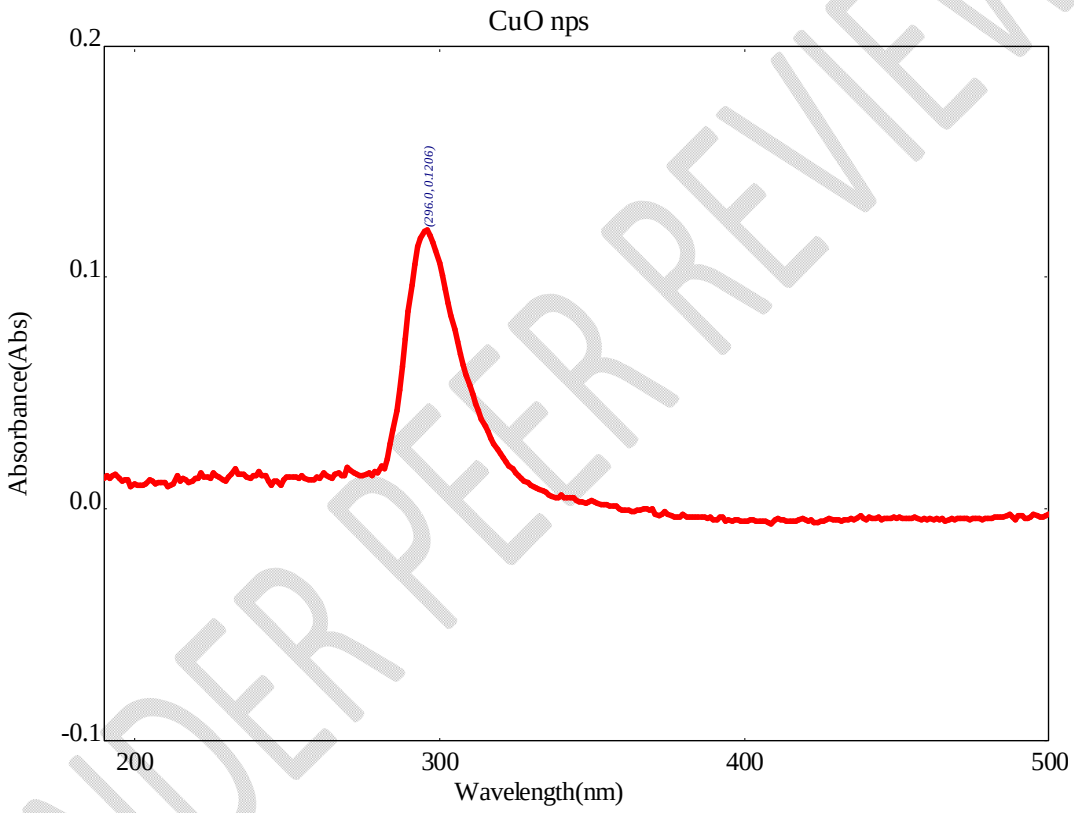

Fig. 1. UV-visible spectra of *Tecoma stans* mediated copper oxide nanoparticles

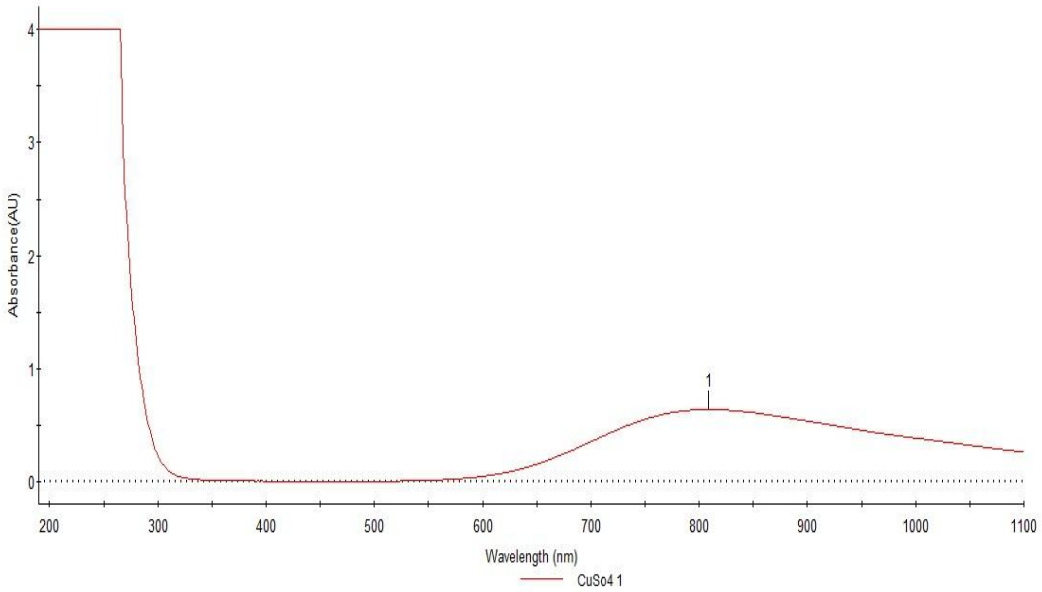

Fig. 2. UV-visible spectra of *reaction mixture of leaf extract of Tecoma stans with copper sulfate* Solutions

3.2 FT-IR Analysis

FTIR spectrums of CuONPs, confirming functional group from plant extract capping the CuONPs. FTIR spectrum of copper oxide nanoparticles was examined to identify the potential bioactive compounds responsible for capping and efficient stabilization of copper oxide nanoparticles synthesized from leaves extract.

FT IR spectra of copper oxide nanoparticles synthesized using green methods are shown in Fig. 3. The spectra shows band at 584.63cm^{-1} corresponding to presence of C-Cl stretch in an organic molecule containing a chloro substituent. Chlorine atoms typically show absorption in the range of 500-600 cm-^{1}. The peak at 932.69 cm-1 in an FTIR spectrum may correspond to C-H and N-H bending vibration in aromatic compounds. The peak at 1019.64cm^{-1} often observed in the IR spectra of ethers such as ethyl ether, methyl tert-butyl ether, and diethyl ether. The C-O stretching vibration occurs at around 1000-1100 cm^{-1}, and the exact frequency depends on the identity of the other atoms in the molecule. The peak at 1280.99cm corresponds to C-N stretching vibration in amines, particularly primary and secondary amines. The band at 1493.95 cm-1 arises from the C=C stretching vibration in the double bond. The peak observed in the IR spectra at 1384.55 cm-1 shows the organic molecules containing methyl and/or methylene groups, where it arises from the C-H bending vibration in these groups. The peak at 1417.94 cm-^{1} shows C-H bending vibration in methyl groups: band at 3430.41cm^{-1} shows the indicates stretching vibration of O-H bond in various organic molecules such as alcohols, phenols and carboxylic acid. The peak at 2852.57cm^{-1} shows stretching vibration of C-H bonds in various organic molecules, such as alkanes, alkenes and aromatic compounds.

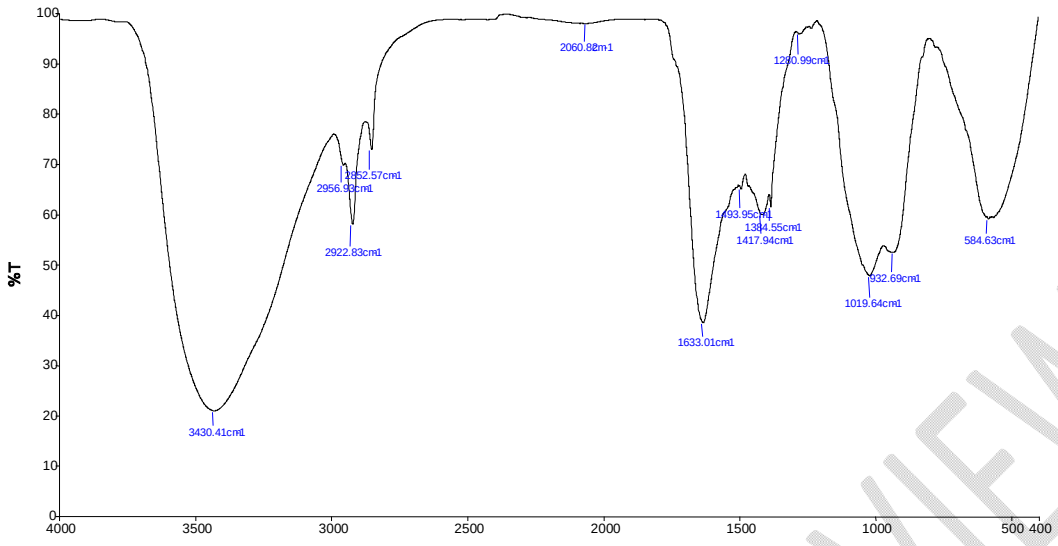

Fig. 3. FT-IR Analysis of *Tecoma stans* mediated copper oxide nanoparticles.

3.3 Scanning Electron Microscope

The surface morphology of the nanoparticles was taken by Scanning Electron Microscopy (SEM) analysis. Scanning Electron Microscopy gives further insight into the morphology and size details of the copper nanoparticles. The SEM photograph shows that the synthesized Copper nanoparticles are asymmetrical dispersed and aggregated infrequently to form free crystal structures. The Surface Morphology of synthesized copper nanoparticles from *T.Stans* leaves extract is shown in Fig.4. Two SEM images were presented, with one image displaying CuO NPs of size 2µm and the other displaying CuO NPs of size 200nm. The particles were observed to have a mixture of shapes, including spherical, hexagonal, and uneven shapes. This information suggests that the morphology of CuO NPs can vary based on their size, and that the particles have a diverse range of shapes.

However, it was not possible for us to investigate the exact surface texture of the observed nanoparticles. Copper nanoparticles prepared by this method show nearly monodispersed distribution of particle sizes, with some traces of lumped particles observed forming bulky micron sized aggregates.

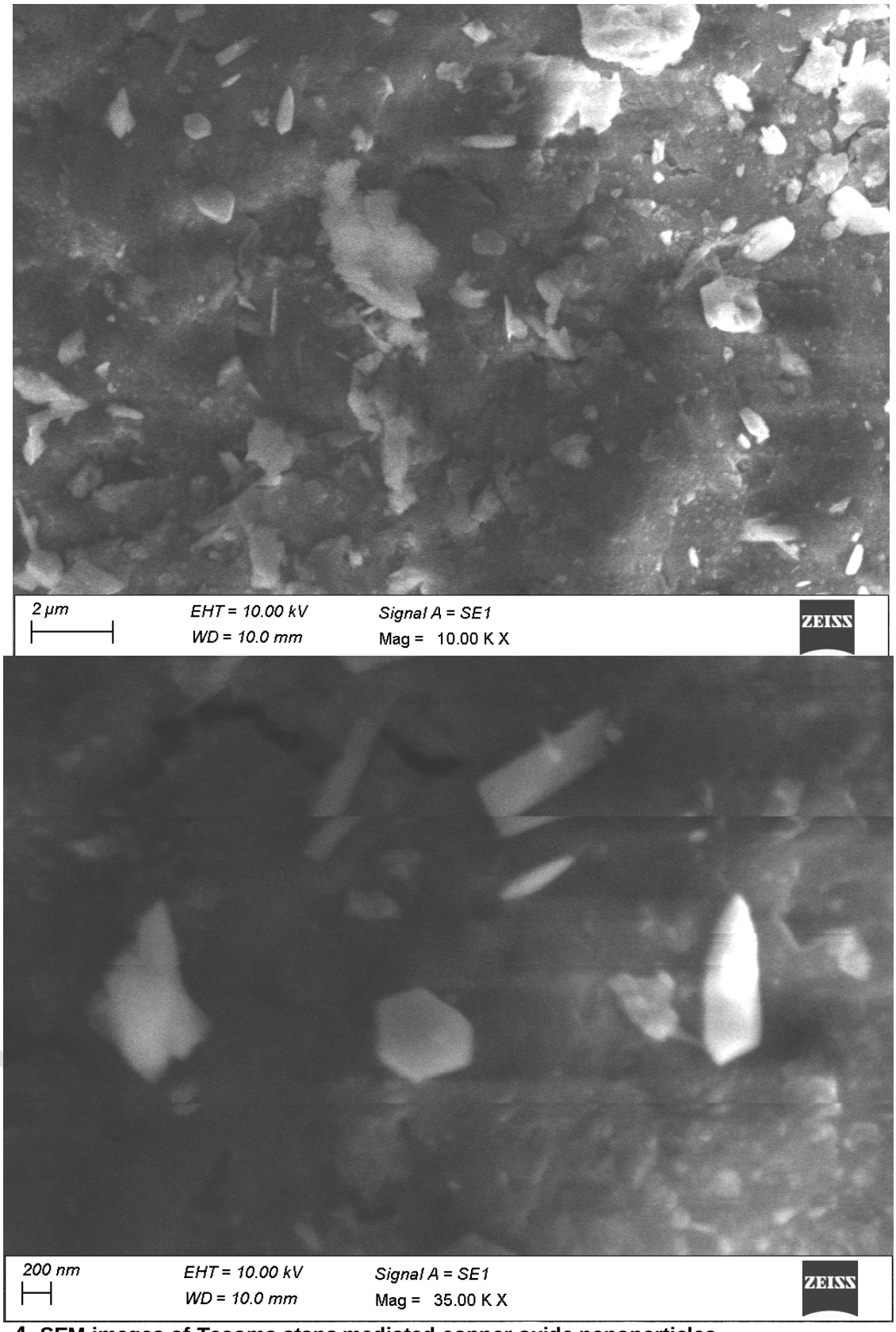

Fig. 4. SEM images of *Tecoma stans* mediated copper oxide nanoparticles

3.4 X-ray Diffraction Analysis

Fig. 4 illustrates the X-ray diffraction (XRD) analysis results of copper oxide nanoparticles synthesized using green methods. The XRD spectra of CuO nanoparticles show two significant peaks at 17°2θ and 32°2θ. The peak at 32°2θ is a characteristic peak of CuO and corresponds to the (002) plane reflection of CuO nanoparticles. It confirms the formation of CuO nanoparticles and provides information about the size and crystallinity of the particles based on the intensity and width of the peak. Similarly, the peak at 17°2θ in the XRD spectra of CuO nanoparticles also corresponds to the (002) plane of CuO crystal structure and is usually sharp and strong, indicating the crystalline nature of the nanoparticles. To calculate the average particle size using X-ray powder diffraction data, we can use the Scherrer equation:d=0.9λ/βcos(θ),where d is the average particle size, λ is the wavelength of X-rays (1.54 Å for Cu Kα radiation), β is the FWHM (Full Width at Half Maximum) of the peak, and θ is the diffraction angle(22). The XRD pattern showed the average particle size of 27.67 Å.

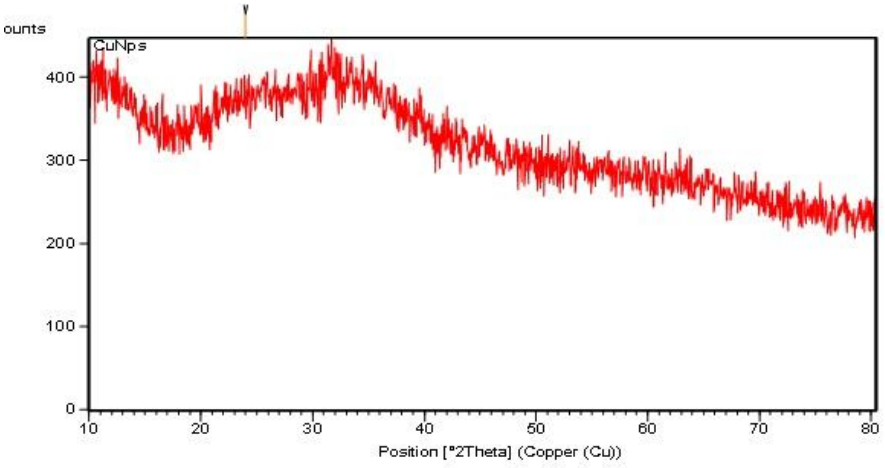

Fig. 5. X-Ray diffraction Analysis of *Tecoma stans* mediated copper oxide nanoparticles

4.CONCLUSION

Green Synthesis of CuO nanoparticles was carried out by using *Tecoma stans* leaves. The characterization was carried out using various analytical techniques including UV-visible spectroscopy, FT-IR analysis, Scanning Electron Microscope, and X-ray Diffraction analysis. The results indicate the successful synthesis of CuO nanoparticles and provide detailed information about their morphology, size, and crystallinity.

The UV-visible spectrum shows strong absorbance of UV rays between the wavelength of 200-300 nm, indicating the presence of CuO nanoparticles in the sample. The peak formed at 398.80 nm is due to surface plasmon absorption of CuO nanoparticles. The FT-IR analysis confirmed the presence of bioactive compounds responsible for capping and efficient stabilization of copper oxide nanoparticles synthesized from leaves extract. The Scanning Electron Microscope analysis showed that the

synthesized Copper nanoparticles are asymmetrical dispersed and aggregated infrequently to form free crystal structures.

The X-ray Diffraction 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 the average particle size of 27.67 Å.

Overall, the characterization results indicate that the synthesized CuO nanoparticles have a diverse range of shapes and sizes, which can 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.

5.CONSENT

It is not applicable.

6.ETHICAL APPROVAL

It is not applicable.

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