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Bio-Reductive Synthesis and Characterization of Copper Oxide Nanoparticles (CuONPs) Using *Alternanthera sessilis* Linn. Leaf Extract

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ABSTRACT

Objectives: The aim of present study was to synthesize and characterize copper oxide nano particles (CuONPs) from leaf extract of *Alternanthera sessilis* (Linn). **Materials and Methods:** Green synthesis of copper oxide nanoparticles has been carried out from aqueous copper sulphate using leaf extract of *Alternanthera sessilis* (Linn.). The bio-molecules present in the leaf juice react with copper sulphate, upon reduction at room temperature resulted in the formation of copper oxide nanoparticles (CuONPs). **Results:** Synthesized nanoparticles (NPs) were studied by SEM- EDAX to investigate the morphology. NPs were spherical and the size in the range of 22.6nm to 25.2nm. The EDAX spectrum revealed the presence of copper oxide. FT-IR spectrum examined the occurrence of bioactive functional groups required for the reduction method. The elemental composition of synthesized product was determined by XRF technique. The oxidation state of NPs was confirmed through the XPS spectra. **Conclusions:** This room temperature, one-pot green synthesis shows promising results for producing CuO nanoparticles in a low cost, eco- friendly process. Metal nanoparticles with antimicrobial activity when embedded and coated on to surfaces can find immense applications in water treatment, synthetic textiles, biomedical and surgical devices, food processing and packaging.

KEYWORDS: CuONPs, reductant, SEM, XPS, XRF

1. INTRODUCTION

Copper oxide(CuO) nanoparticles are prominent due to their varied applications in superconductors, optical[1], electrical[2], nanofluids[3], catalytic[4,5], photocatalytic degradation[6], gas sensors, and in biosensors.[7] CuO is an antimicrobial, anti-biotic and anti-fungal agent when incorporated in coatings, plastics, textiles, etc.[8] It acts as a catalyst to eliminate industrial effluent in the environment. The elimination of these pigments and other organic materials ensures a safe and clean environment.[9]

The preparation of CuONps uses a variety of methods including sol-gel, quick precipitation, sono chemical, electrochemical, solid state reaction and microwave irradiation. Therefore it was challenge to find a convenient, mild, non-toxic natural product to produce metal nanoparticle in an aqueous environment. Hence the need to develop, newer eco-friendly processes for the synthesis of metal/metal oxide nanoparticles of controlled shapes and sizes, could permit them to be extensively used in biological system. Green synthesis is proved a best candidate for nanoparticle synthesis using plants, which are more stable with faster rate of production. Copper oxide nano structures can be synthesized from variety of precursors such as cupric sulphate[10,11], cupric chloride[12], cupric acetate[13] etc.

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Alternanthera sessilis (Linn.)R.Br. belongs to Amaranthaceae family, a perennial herb, bearing short petiole, simple leaves with white flowers found throughout in India. It is known in English as sessile joyweed or dwarf copperleaf. The plant is traditionally used for treatment of skin diseases, haemorrhoids, liver and spleen disorders, asthma, wound healing[14] and acts as anti-diabetic drug.[15,16] The major chemical constituents include alkaloids, flavonoids, phenols, beta carotene, sterols, proteins, carbohydrates and ascorbic acid.[17] Bio-active components in the leaf extract may act as a vehicle for the preparation of nanoparticles.

The present work reports the synthesis of copper oxide nanoparticles using the leaf extract of *A.sessilis Linn*. The characterization was done by scanning electron microscope (SEM) EDAX, Fourier transform infrared spectroscopy (FT-IR), X-ray fluorescence (XRF) and X-ray photoelectron spectroscopy (XPS) analysis. The procedure involves self-sustained reaction in homogeneous solution of copper sulphate and *A. sessilis Linn* leaf extract.

2. Experimental

2.1 Chemicals

All reagents used in the study were of analytical grade. Copper sulphate used was purchased from Qualigens Fine Chemicals.

2.2 Collection of plant samples

Leaves of *Alternanthera sessilis L* were purchased from local market, Thanjavur-Tamilnadu, India. The leaves were identified and authenticated by Dept. of CARISM, SASTRA University, Thanjavur.

2.3 Extract preparation

100g of fresh leaves of *Alternanthera sessilis L.* was macerated with 750ml of distilled water. The mixture was frequently shaken for 8hours and allowed to stand for 16 hours, filtered through whatman no.1 filter paper. The filtrate was collected in a clean conical flask and used for the nanosynthesis.

2.4 Bio-green synthesis of copper oxide nanoparticles

For the synthesis of nanoparticles, 20ml of leaf extract was mixed with 80ml of 1mM copper sulphate solution. The mixture was kept for incubation at room temperature in dark. The colour changed from brown to black indicating the formation of copper oxide nanoparticles. The nanoparticles thus obtained were purified by repeated centrifugation at 7000rpm for 10min. The pellet was collected and dried. The schematic formation of Cu (O) nanoparticles by reduction method was shown in (Fig 1).

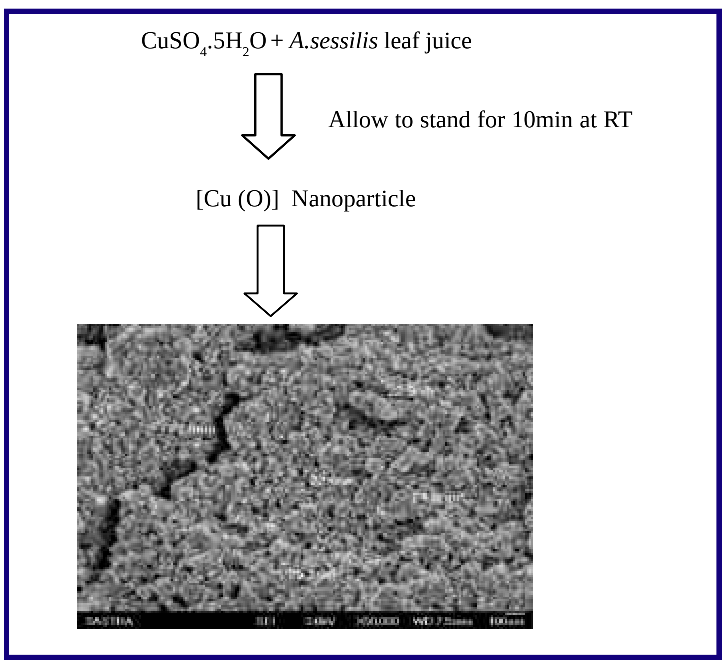

Fig 1: Schematic formation of Cu (O) nanoparticles by reduction method

2.5 Characterization of copper oxide nanoparticles

Morphological features of synthesized nanoparticles were studied by FE-SEM with EDAX (JEOL JSM 6701-F). FTIR analysis was carried out by Perkin Elmer spectrometer (Spectrum one)ranging from 4000cm^{-1}- 400cm^{-1}.The elemental composition of resulted nanoparticles was studied using XRF analysis. Oxidation state of CuONPs was analyzed through XPS (K-Alpha, Thermo scientific, and USA).

3. RESULTS AND DISCUSSION

FTIR measurements were carried out to identify the possible biomolecules responsible for the reduction and capping of the efficient stabilization of the bio reduced copper nanoparticles. Fig 2 shows an absorption peak of 1639 cm^{-1} may be assigned to the amino group of protein and peak at 3429 cm^{-1} is assigned to -OH stretching of phenolic compounds**.** The proteins present in the synthesized nanoparticles have been found to be effective stabilizing agent by forming a coating on the surface of the nanoparticles. Many biomolecules present in plat extract like alkaloids, phenols, proteins, carbohydrates, and ascorbic acid are found to be involved in bioreduction, formation and stabilization of metal nanoparticles.[18] The absorption peak at 1037cm^{-1} relates to C-N vibrations. There is a peak observed at 617cm^{-1} in the spectrum which is the characteristics of Cu-O bond formation.

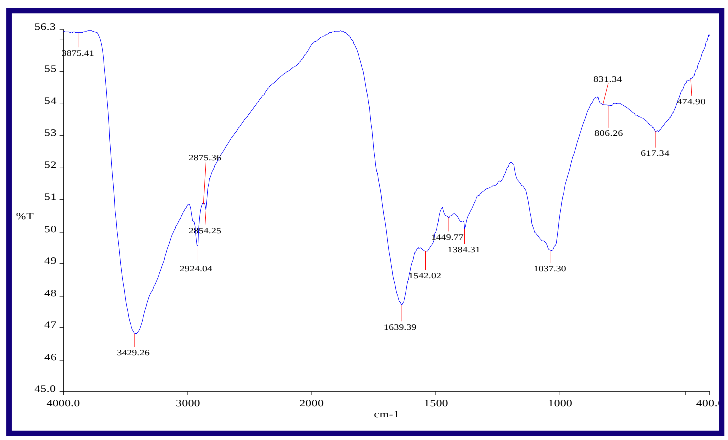

Fig 2: FTIR spectra of CuONPs from A.sessilis leaf extract

The morphological features of biosynthesized CuO nanoparticles were shown in SEM image (Fig 3a). It is evident from that the nanoparticles are of spherical in shape and size in the range of 22.6 to 25.2nm. EDAX spectrum showed the presence of elemental copper oxide along with impurities (Fig 3b).

Elemental composition of nanoparticle was determined by Energy dispersive X-ray Fluorescence (XRF) analysis. . The maximum percentage of elemental copper oxide was found to 75.78% along with trace level of other elements.

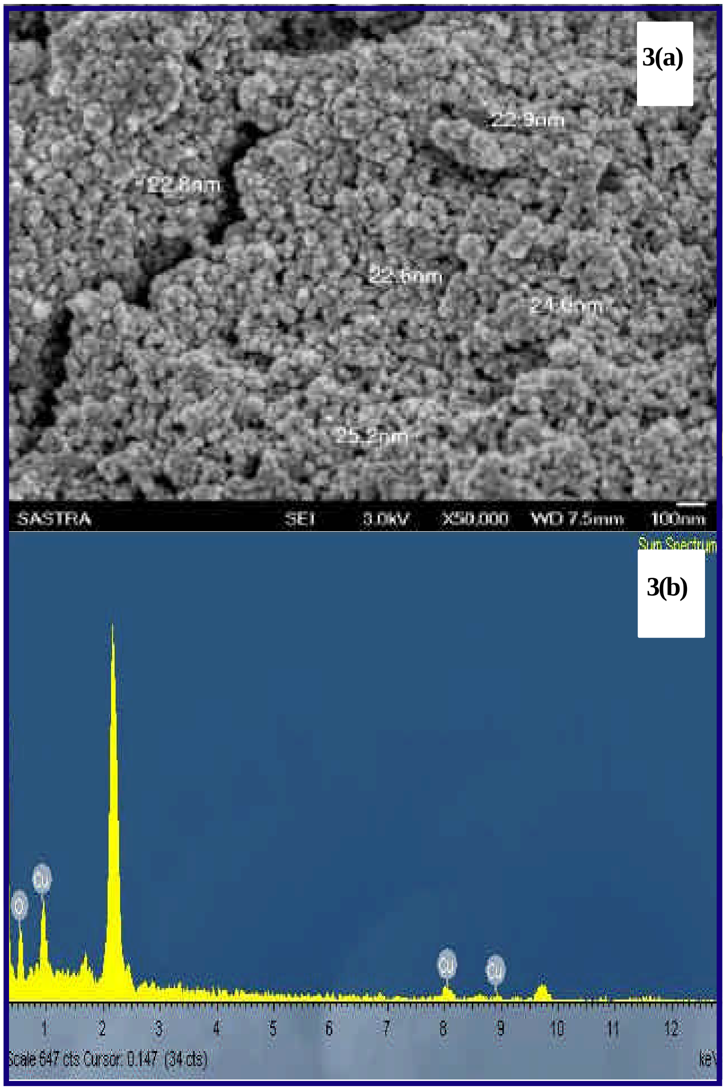

Fig 3(a, b): SEM images and EDAX spectrum of CuO nanaoparticles

The composition of NPs was analyzed by oxide mode of X-ray fluorescence spectrometer.

The elemental composition and oxidation state of CuO nanoparticles were analyzed through XPS technique. The survey scan recorded for the sample shows the presence of Cu and O (Fig 4a). A selected area scan is also recorded for the individual elements such as Cu2p shown in Fig 4b. Cu2p core level spectrum represents the two peaks located at 932.7 and 933.1eV which correspond to the Cu2p$_{3/2}$. This value match well with the data reported for the copper in copper oxide. In addition to that the satellite peak of Cu2p located at 943 and 945eV distinguish the oxidation state of copper (Fig 4c.). The O1s core level spectrum was shown in Fig 4d. The peak at the low binding energy value of 529eV is due to the oxygen in CuO which corresponds to the O-Cu bond. Other peaks also located at 285 and 399.8eV respectively. This result is in good agreement with previous literature data.[19]

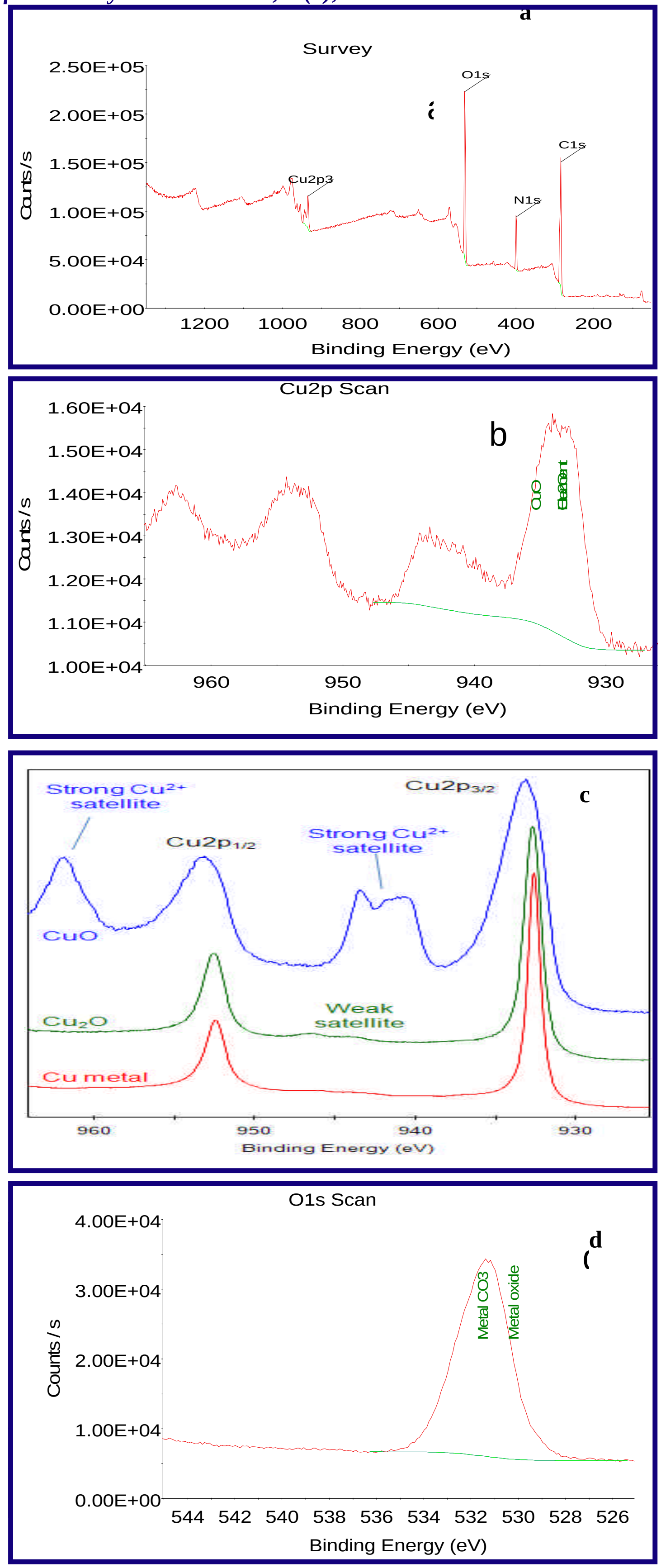

Figure 4: XPS spectra of (a) survey scan (b) Cu2p scan (c) core level spectrum of Cu2p3/2 (d) O1s scan

4. CONCLUSION

Here we described an appropriate method for the synthesis of CuONPs using bioreduction. It is the most frequently applied method for the preparation of nanoparticles. This is a simple, green and excellent method for the synthesis of CuONPs at RT without using any toxic chemicals. For the production of nanoparticles, *A. sessilis* extract is a rapid method due to it's bio-active constituents.In future, green synthesis can be scaled up to prepare large quantities for their extensive application for the degradation of organic pollutants, bioremediation systems, brain cancer, cytotoxicity and drug delivery.

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