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Sarcostemma acidum stem extract mediated synthesis and characterization of copper oxide nanoparticles

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Abstract
Metal oxide nanoparticles (NPs) are receiving significant consideration for possible applications in optoelectronics, nano devices, nano sensors, information storage and nano electronics. The aim of the study is to investigate the preparation and characterization of CuONPs using extract of *Sarcostemma acidum*. *Sarcostemma acidum* extract has been found to be suitable for the synthesis of copper oxide nanoparticles through green method. Spherical, polydispersity of CuONPs of particle sizes ranging from 32 – 82nm are obtained. Changes of color occur due to surface plasmon resonance during the reaction with the ingredients present in the extract of *Sarcostemma acidum* extract, resulting in the formation of CuO nanoparticles. It is confirmed by FT-IR, UV–Visible spectroscopy and SEM analysis. FT-IR spectroscopic study confirm that the phenol, alcohol, alkynes and aromatics feature a strong binding ability with oxide, suggesting the formation of a layer covering oxide nanoparticles and acting as a capping agent to stop agglomeration and supply stability to the medium. The possible biomolecule like phenol, alcohol, alkynes and aromatics are responsible for the bio reduction process.

Keywords: *Sarcostemma acidum*, Synthesis and characterization, Copper oxide nanoparticles (CuONPs)

Introduction

In this era, nanotechnology is one of the greatest attention-grabbing areas which is used to describe the creation and utilization of materials with structural features between those of atoms and bulk materials with tiniest dimension in the nano range (Brunner *et al*. 2006) [1]. Metal oxide nanoparticles (NPs) are receiving significant consideration for possible applications in optoelectronics, nano devices, nano sensors, information storage and nano electronics (Marabelli, 1995) [2]. Among numerous metal oxide nanoparticles, copper nanoparticles have excellent chemical and physical properties. Copper nanoparticles have extensive applications as antimicrobial materials, sensors and catalysts. They are super strong materials used in heat transfer systems. Copper nanoparticles are highly reactive due to their high surface-to-volume ratio and may easily interact with other particles (Narayanan *et al*., 2003) [3].

Processes used for nanoparticles synthesis are physical, chemical and a currently developed biological method. Chemical methods have numerous drawbacks including the generation of hazardous by-products, utilization of toxic solvents and high energy consumption, which pose hazards to human health and to the environment. Therefore, the biological method has a plus over physical and chemical methods of nanoparticle synthesis, because it is environmentally friendly and cost-effective (Nabhikha *et al*. 2009) [4]. The major biological materials involved during this are fungi, bacteria and plant mediated extracts. In current years, the synthesis of nanoparticles using plant mediated extracts has gained more attention. The synthesis and applications of copper nanoparticles has been studied by many researchers using several plants (Swarnkar *et al*., 2009; Abdul Rahman *et al*., 2009; Ansilin *et al*., 2016) [5-7]. Hence in the present study, the synthesis and characterization of CuONPs using extract of *Sarcostemma acidum* has been carried out.

Materials and Methods

Plant materials

The stem of *Sarcostemma acidum* were collected from Thanjavur, District, Tamil Nadu, India. The *Sarcostemma acidum* were first washed well and dust was removed from the stems. Then the stems were dried at room temperature and coarsely powdered.

Preparation of extract

Sarcostemma acidum aqueous extract was prepared by placing 10 g of dried fine powder in 500 ml glass beaker along with 400 ml of sterile distilled water. The mixture was boiled for 10 min until the colour of solution changed from watery to brown-yellow. Then the mixture was cooled to room temperature and filtered with Whatman No. 1 paper, before centrifuging at 1200 rpm for five minutes to get rid of biomaterials. The extract was stored at room temperature in order to be used for further experiments.

Copper oxide nanoparticle (CuONPs) synthesis

In the synthesis of copper oxide nanoparticles, 2.8g of copper acetate monohydrate was dissolved in 500 ml of the deionized water and stirred magnetically at room temperature for 5 minutes. *Sarcostemma acidum* stem aqueous extract were added dropwise under magnetic stirring and the blue color of copper ions change to green color, when the extract comes in contact copper ions. The acquired green mixture was kept at room temperature. After 10 minutes, the green mixture started altering to a brown suspended mixture, representing the development of water soluble monodispersed copper oxide nanoparticles (Ghidan *et al*., 2016) [8].

Characterization of Nanoparticles

UV and FTIR Spectroscopic analysis

The reduction of pure Cu^{+} ions was examined under visible and UV light for proximate analysis. For UV and FTIR spectrophotometer analysis, the extracts were centrifuged at 3000 rpm for 10 minutes and filtered through Whatmann No. 1 filter paper by using high pressure vaccum pump. The sample is diluted to 1:10 with the same solvent. The reduction of pure Cu^{+} ions were scanned in the wavelength ranging from 200-800 nm using Perkin Elmer Spectrophotometer and the characteristic peaks were detected. FTIR analysis was performed using Spectrophotometer system, which was used to detect the characteristic peaks ranging from 400-4000 cm^{-1} and their functional groups. The peak values of the UV and FTIR were recorded. Each and every analysis was repeated twice for the spectrum confirmation.

Electron microscopy and EDX analysis of copper nanoparticles

In this research work, ZEEISS-SEM machine was used to characterize mean particle size, morphology of nanoparticles. The freeze dried sample of CuONPs solution was sonicated with water, small drop of this sample was placed on glass slide allowed to dry. A skinny layer of platinum was coated to form the samples conducive. ZEEISS-SEM machine was operated at a vacuum of the order of 10-5 torr. The accelerating voltage of the microscope was kept within the range 10 kV. Compositional analysis on the sample was administered by the energy dispersive X-ray spectroscopy (EDX) attached with the SEM. The EDX analysis of Cu sample was done by the SEM machine

Results and Discussion

Synthesis of copper nanoparticles

Phytosynthesis of Cu nanoparticles by the aqueous extract of *Sarcostemma acidum* stem was carried out in this work. During the visual observation, copper acetate and stem extract stirred magnetically showed the green mixture started changing to a brown suspended mixture after 10 min. When the extract comes in to contact with copper ions, there is a spontaneous change from the blue color of copper ions to green color, which is clear indication for the formation of water soluble monodispersed copper oxide nanoparticles (Figure 1).

Fig 1: Colour changes before (Plant extract) and after (CuONPs), the process of reduction of Cu^{+} to Cu nanoparticles and control (Copper acetate)

Plant = *Sarcostemma acidum* extract only
Copper acetate = Copper acetate without *Sarcostemma acidum* extract.
CuONPs = CuONPs with *Sarcostemma acidum* extract and copper acetate after 5 hrs of incubation (Green colour)

UV-VISIBLE spectral analysis

It is generally recognized that UV–Visible spectroscopy could be used to observe size and shape-controlled nanoparticles in aqueous suspensions. Figure 2 shows the UV-Visible spectra recorded from the reaction medium. In the UV–Visible spectra of the reaction mixture of copper acetate solution with *Sarcostemma acidum* extract, the peak was observed at 282nm indicating the reduction of copper acetate monohydrate into CuONPs. It was also observed that the reduction of copper acetate ions into copper oxide nanoparticles was completed at 10 minutes in room temperature, indicating rapid biosynthesis of copper oxide nanoparticles. The peak was elevated due to inter-band transition of core electrons of CuONPs in the reaction combination and the increase of peak specified that the particles are polydispersed. The absorbance wave length values are closely matched with the reported values (Ghidan *et al*., 2016; Swarnkar *et al*., 2009; Abdul Rahman *et al*., 2009) [8,5,6].

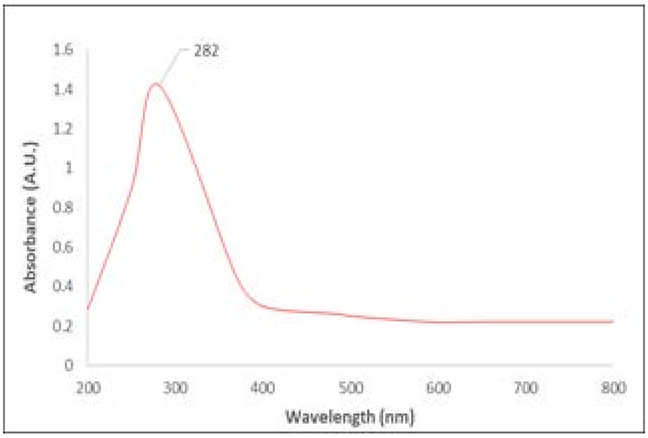

Fig 2: UV-Visible Spectral analysis of CuONPs Fourier Transform Infra-Red spectral analysis of CuONPs

FTIR spectrum of copper nanoparticles was examined to identify the possible biomolecules, responsible for capping and efficient stabilization of the copper nanoparticles synthesized by plant leaves extract. The peaks observed (Figure 3) for phytochemicals capped copper nanoparticles formed through reduction by *Sarcostemma acidum* stem at 3444.81cm^{-1} (Alcohol, Phenol), 1633.61cm^{-1} (1˚amines), 1271.12cm^{-1} (Aromatic amines), 1114.01cm^{-1} (Aliphatic amines) and 665.85cm^{-1}(alkenes) suggest the presence of flavonoids and phenols adsorbed on the surface of Cu nanoparticles. The results of FTIR analysis confirmed the presence of alcohol, phenol, aromatics amines, 1˚amines, aliphatic amines and alkynes. This confirms the presence of secondary metabolites in the sample as proposed capping and stabilizing agent.

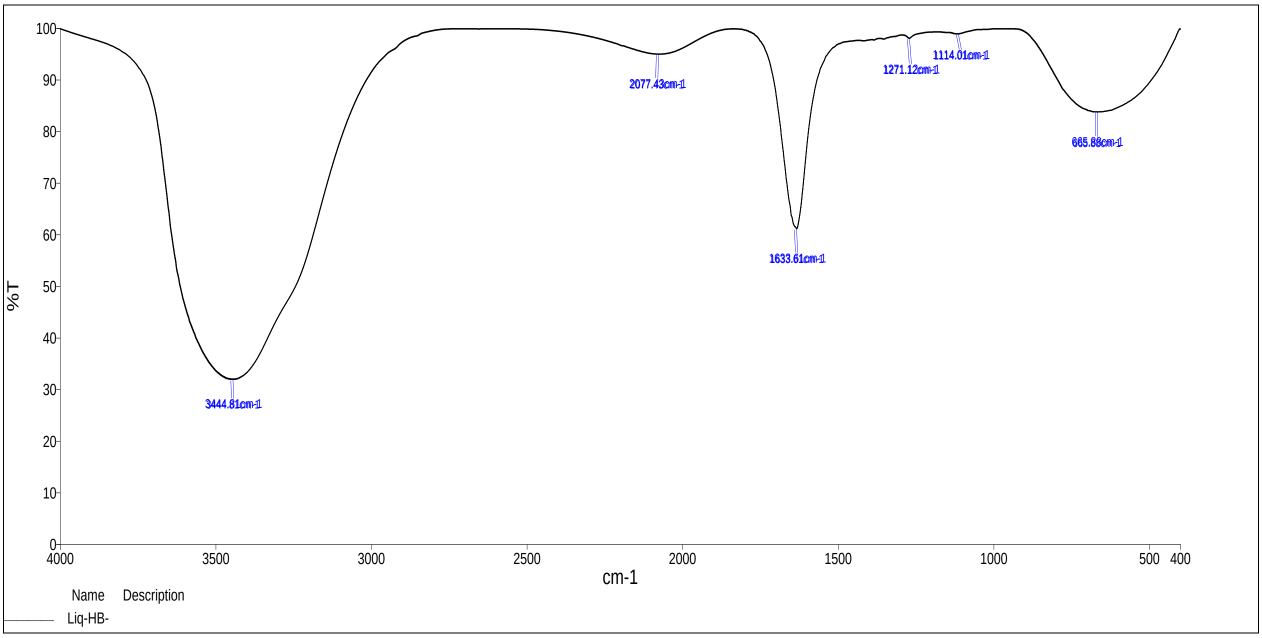

Fig 3: FTIR spectrum of Copper nanoparticles synthesized by reduction of Cu^+ ions by *Sarcostemma acidum* stem extract.

Scanning Electron Microscope (SEM) analysis of CuONPs

SEM and TEM analysis were used to study the size of the copper oxide nanoparticles, which showed the synthesis of CuONPs with higher density polydispersed sphericals of various sizes. SEM analysis was carried out to understand the topology and the size of the NPs, which showed the synthesis of higher density polydispersed spherical NPs of various sizes that ranged from 30 to 80nm. Most of the nanoparticles grouped and only a few of them were dispersed, when observed under SEM (Figure 4).

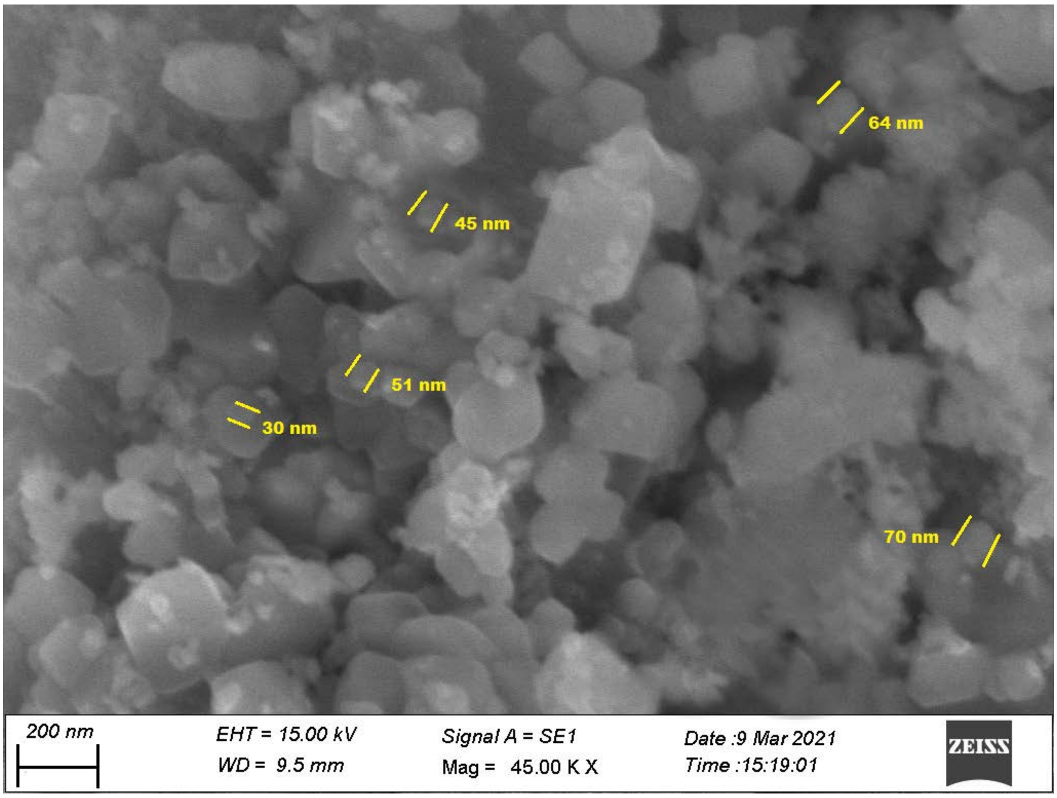

Fig 4: Copper nanoparticles (CuONPs) viewed in scanning electron microscopic (SEM) ranged between 30 – 80nm

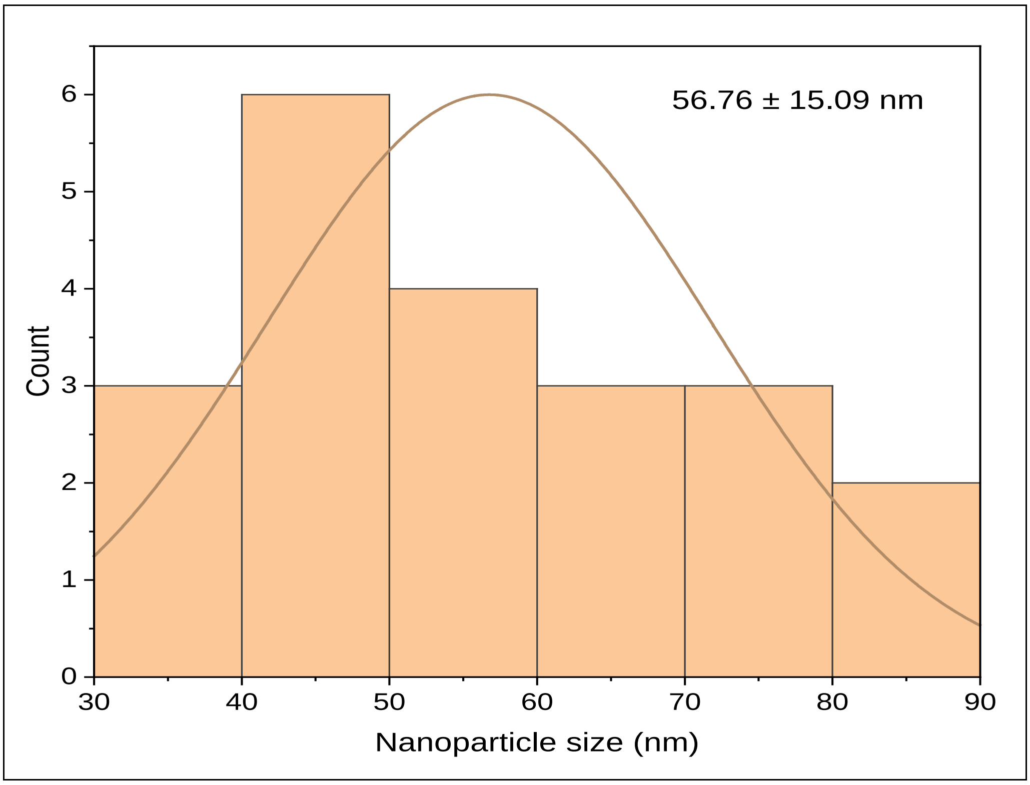

Fig 5: Histogram of Copper nanoparticles (CuONPs) ranged between 32 – 82nm using SEM image

The histograms (Figure 5) plotted on the obtained data to study the particle size distribution reveals that the size of the nanoparticles ranged from 32 to 82 nm and the average particle size was found to be 56.76 ± 15.09 nm. Overall Particle size of NPs were highly distributed between 40nm to 60nm which is the evidence that the NPs synthesized less than 100nm (NPs < 100nm).

Energy-dispersive X-ray spectroscopy (EDX) analysis of CuONPs

The data that is created by EDX examination consists of spectra with peaks consistent to all the different elements that are present in the sample. EDX of CuONPs revealed the presence of pure copper (Cu 50.72%) and was the major constituent element compared to oxygen (38.33%) and carbon (10.95%) as shown in Fig 6. The EDX reading proved that the required phase of copper (Cu) was present in the CuONPs.

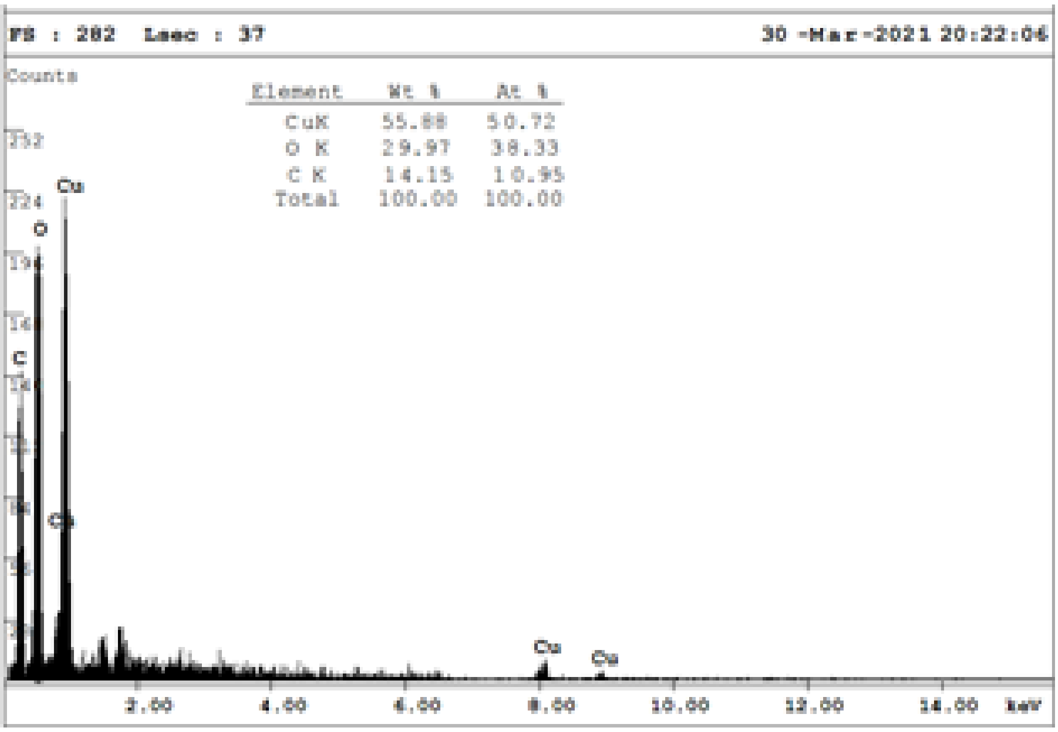

Fig 6: EDX -Spectroscopy view of the elemental composition of CuONPs indicated higher percentage of copper signal

Present study agreement with earlier reports showed the Copper oxide nanoparticles have been synthesized and characterized by using plants extract of leaves, fruits, roots, seeds, barks, stem, peels and flowers such as *Caesalpinia bonducella* (Sukumar *et al*., 2020) [9], *Enicostemma axillare* (Lam.) (Mali *et al*., 2019) [10], *Azadirachta indica (*Rafique *et al*., 2018) [11], *Cassia auriculata* (Prasad *et al*., 2017) [12], *Punica granatum* (Ghidan *et al*., 2016) [8] and *Malva sylvestris* (Awwad *et al*., 2015) [13].

Conclusion

Green synthesis of copper oxide nanoparticles CuONPs is an eco-friendly and safer to environment as compared with chemical and physical methods. We have developed a fast, eco-friendly, and convenient green method for the synthesis of CuO nanoparticles from copper acetate monohydrate using *Sarcostemma acidum* stem aqueous extract. *Sarcostemma acidum* stem extract was found suitable for the green synthesis of copper oxide nanoparticles at ambient conditions. Spherical, polydispersity of CuONPs of particle sizes ranging from 30 to 80nm are obtained. Color changes occur due to surface plasmon resonance during the reaction with the ingredients present in the *Sarcostemma acidum* stem extract resulting in the formation of CuO nanoparticles, which is confirmed by FT-IR, UV–vis spectroscopy EDX and SEM. FT-IR spectroscopic study confirmed that the phenol, alcohol, alkynes and aromatics features a strong binding ability with oxide, suggesting the formation of a layer covering oxide nanoparticles and acting as a capping agent to stop agglomeration and supply stability to the medium, the possible biomolecule like phenol, alcohol, alkynes and aromatics responsible for the bioreduction process.

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