



Synthesis and characterization of copper oxide nanoparticles using *Dictyota dichotoma* extract

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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 was to investigate the preparation and characterization of CuONPs using extract of *Dictyota dichotoma*. *Dictyota dichotoma* extract was found suitable for the synthesis of copper oxide nanoparticles through green method. Spherical, polydispersity of CuONPs of particle sizes ranging from 12 to 52 nm are obtained. Changes of color occur due to surface plasmon resonance during the reaction with the ingredients present in the extract of *Dictyota dichotoma* resulting in the formation of CuO nanoparticles, which is confirmed by, FT-IR, UV-vis spectroscopy 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.

Keywords: *Dictyota dichotoma*, synthesis and characterization, copper oxide nanoparticles (CuONPs)

Introduction

In this era, nanotechnology is one of the greatest attention-grabbing areas which are used to describe the creation and utilization of materials with structural features between those of atoms and bulk materials with at tiniest one 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 are excellent chemical and physical properties and low price of preparation and also great interest. Copper nanoparticles have extensive applications as antimicrobial materials, heat transfer systems, sensors, super strong materials and catalysts. Copper nanoparticles are fast 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 (Prabhu and Poulouse 2012) [5]. In current years, the synthesis of nanoparticles using plant mediated extracts has attention more importance. The synthesis and applications of copper nanoparticles studied by many researchers using several plants (Swarnkar *et al.*, 2009; Abdul Rahman *et al.*, 2009; Ansilin *et al.*, 2016) [6, 7, 8]. Hence in this study, for the first

time we evaluated the synthesis and antimicrobial activity of CuONPs using extract of *Dictyota dichotoma*.

Materials and Methods

Preparation of leaves extract

Whole plant of *Dictyota dichotoma* were collected from Mandapam, Ramanathapuram District, Tamil Nadu. The plant authenticated by Ministry of Environment, forest, and Climate Change, Botanical survey of India, Southern Regional Centre, TNAU campus, Lawley Road, Coimbatore. The identified specimen (Ref No.: BSI/SRC/5/23/2019/Tech./2934) was preserved in our department. Whole plant was washed several times with distilled water to remove dust particles and then dried in shade for two weeks to remove the residual moisture.

Preparation of extract

Dictyota dichotoma 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 temperature and filtered with Whatman No. 1 paper before centrifuging at 1200 rpm for five min 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 a synthesis of copper oxide nanoparticles, 2.8 g of copper acetate monohydrate was dissolved in 500 ml of the deionized water and stirred magnetically at room temperature for 5 min. *Dictyota dichotoma* leaves aqueous extract were added dropwise under magnetic stirring and change the blue color of copper ions to green color when the leaves extract comes in contact copper ions. The acquired green mixture was kept at room temperature. After 10 min,

the green mixture started altering to a brown suspended mixture, representing the development of water soluble monodispersed copper oxide nanoparticles (Ghidan *et al.*, 2016) [9].

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 min and filtered through Whatmann No. 1 paper by using high vacuum pump. The sample is diluted to 1:10 with an equivalent solvent. The reduction of pure Cu⁺ ions were scanned within the wavelength starting from 200-800 nm using Perkin Elmer Spectrophotometer and therefore the characteristic peaks were detected. FTIR analysis was performed using Spectrophotometer system, which was wont to detect the characteristic peaks in starting from 400-4000 cm⁻¹ and their functional groups. The height values of the UV and FTIR were recorded. Each and each analysis was repeated twice for the spectrum confirmation.

Electron microscopy and EDX analysis of copper nanoparticles

In this research work, ZEEISS-SEM machine were wont 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 conductive 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 (EDS) 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 leaves extract of *Dictyota dichotoma* leaves was carried out in this work. During the visual observation, copper acetate and leaves extract stirred magnetically showed the green mixture started changing to a brown suspended mixture after 10 min. The appearance of the leaf extract comes in contact copper ions spontaneous change the blue color of copper ions to green color is clear indication for the formation of water soluble monodispersed copper oxide nanoparticles (Fig 1).

UV-VIS spectral analysis

It is generally recognized that UV-Vis spectroscopy could be used to observe size and shape-controlled nanoparticles in aqueous suspensions. Figure 2 shows the UV-Vis spectra recorded from the reaction medium. In the UV-Vis spectra of the reaction mixture of copper acetate solution with *Dictyota dichotoma* leaves extract the peak was observed at 286nm indicating indicating the reduction of copper acetate monohydrate into CuONPs. It was also observed that the reduction of copper acetate ions into copper oxide nanoparticles started at the start of reaction and reduction

was completed at almost 10 min at 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) [9, 6, 7].

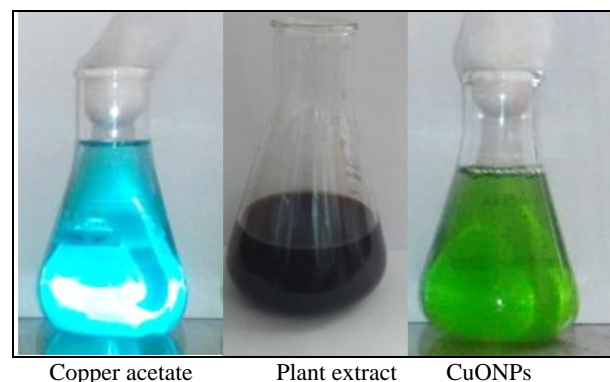


Fig 1: Colour changes before (Plant extract) and after (CuONPs) the process of reduction of Cu⁺ to Cu nanoparticles and control (Copper acetate)

Plant=*Dictyota dichotoma* extract only

Copper acetate= Copper acetate without *Dictyota dichotoma* extract.

CuONPs=CuONPs with *Dictyota dichotoma* extract and copper acetate after 5 hrs of incubation (Green colour)

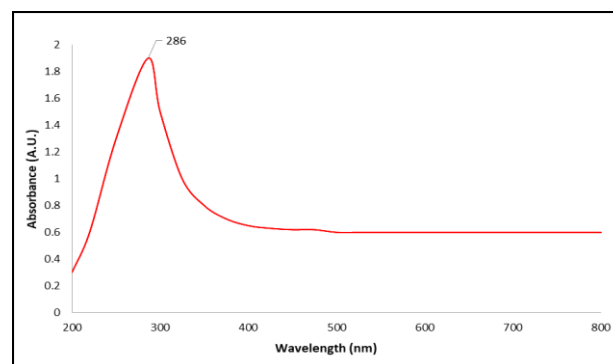


Fig 2: UV-Vis Spectral analysis of CuONPs

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 (Fig. 3) for phytochemicals capped copper nanoparticles formed through reduction by *Dictyota dichotoma* leaves at 3438.79cm⁻¹ (Alcohol, Phenol), 1631.48 cm⁻¹ (aromatic rings) and 621.85cm⁻¹(alkynes), 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 and alkynes compound. This proves the occurrence of phytochemicals in the sample as offered capping and stabilizing agent.

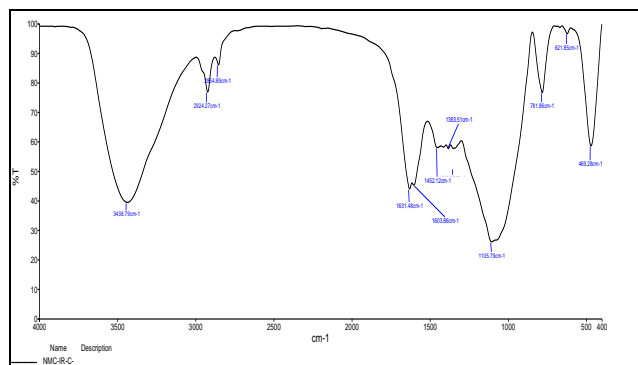


Fig 3: FTIR spectrum of Copper nanoparticles synthesized by reduction of Cu^+ ions by *Dictyota dichotoma* leaves extract.

Scanning and Transmission Electron Microscope (SEM) analysis of CuONPs

SEM and TEM analysis showed to understand the the size of the Copper oxide nanoparticles, which showed the synthesis of CuONPs with higher density polydispersed spherical of various sizes. SEM study showed the various from 12.5 – 52.10nm as crystalline nature and well spherical of the nanoparticles. Most of the nanoparticles grouped and only a few of them were dispersed, when observed under SEM (Fig. 4).

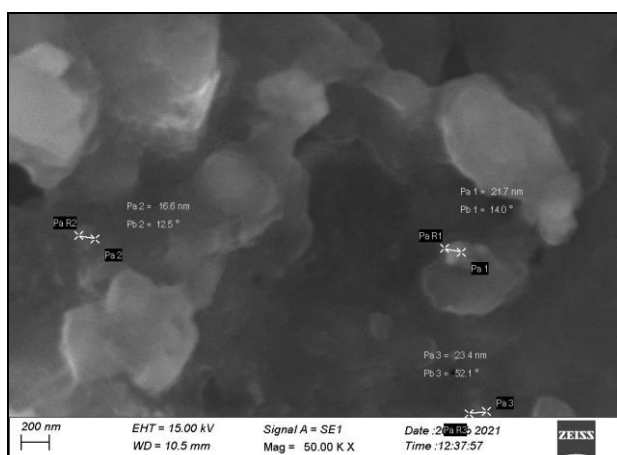


Fig 4: Copper nanoparticles (CuONPs) viewed in scanning electron microscopic (SEM) and ranged between 12.5 – 52.10nm

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

The data that is created by EDX examination

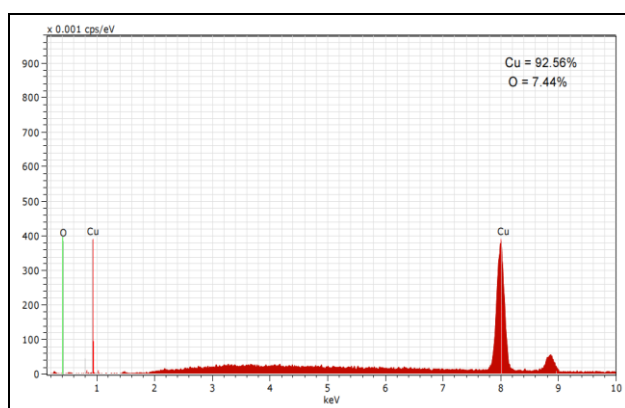


Fig 5: EDS-Spectroscopy view the elemental composition of CuONPs indicated higher percentage of copper signal

Consists of spectra with peaks consistent to all the different elements that are present in the sample. EDS of CuONPs revealed the presence of pure copper (Cu 92.56%) and was the major constituent element compared to oxygen (7.44%) as shown in Fig 5. The EDX reading proved that the required phase of copper (Cu) was present in the CuONPs.

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 *Dictyota dichotoma* aqueous extract. *Dictyota dichotoma* extract was found suitable for the green synthesis of copper oxide nanoparticles at ambient conditions. Spherical, polydispersity of CuONPs of particle sizes ranging from 12 to 52nm are obtained. Color changes occur due to surface plasmon resonance during the reaction with the ingredients present in the *Dictyota dichotoma* 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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