Journal of Nanoscience and Technology 2(5) (2016) 221–223

ISSN: 2455-0191

Contents List available at JACS Directory

Journal of Nanoscience and Technology

journal homepage: www.jacsdirectory.com/jnst

Special Issue on "National Conference on Advanced Materials and Their Applications" *Issue Editor:* Dr. C. Vedhi

Green Synthesis and Characterisation of Copper Oxide Nanoparticles using *Azadirachta indica* (Neem) Leaf Aqueous Extract

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ARTICLE DETAILS

Article history:
Received 22 November 2016
Accepted 12 December 2016
Available online 31 December 2016

Keywords:
Green synthesis
Copper Oxide Nanoparticles
Azardiachta indica (Neem)

ABSTRACT

Development of green technology is generating interest of researchers towards eco-friendly and low cost method for bio synthesis of nanoparticles. 0.1 M copper acetate solution and neem extract was made into bulk. 50 mL of copper acetate and 150 mL of neem extract were taken and mixed in a beaker. It was observed that the *Azardiachta indica* leaf extract can reduce copper ions into copper nano particles within 5 minutes of reaction time and brown coloured precipitate settling down. The biosynthesized nanoparticles of copper oxide are characterized through UV-Vis spectra, FT-IR, AFM and XRD. In UV-Vis spectra a peak was obtained at 346 nm due to inter band transition of core electrons and band gap energy of 2.65 eV was observed through tauc plot. Sharp peak at 480.42 and 538.80 cm^{-1} in FT-IR spectrum is due to Cu-O bond. CuO Nano particle is crystalline and identical to simple cubic. The particle sizes of CuO NPs are found in different sizes range of 49 nm, 87 nm, 120 nm and 324 nm.

1. Introduction

Many physical and chemical methods which have been developed for preparing metallic nanoparticles, nanotechnology also serves as an important method in the development of clean, nontoxic and environmentally friendly procedures for the synthesis and assembly of metallic nanoparticles. Biosynthesis of metallic nanoparticles using plant extract is a fabulous and emerging eco-friendly science of well-defined size, shapes and controlled mono disparity [1-3].

Metal oxide nanoparticles (NPs) have been receiving considerable attention for their potential applications in optoelectronics, nano sensors, nano devices, nano electronics, information storage, and catalysis [4]. Among various metal oxide NPs, copper nanoparticles, due to their excellent physical and chemical properties and low cost of preparation, have been of great interest. Copper nanoparticles have wide applications as heat transfer systems, antimicrobial materials, super strong materials, sensors and catalysts. Copper nanoparticles are very reactive because of their high surface-to-volume ratio and can easily interact with other particles [5] and increase their antimicrobial efficiency.

In literature, the Cu nanoparticles are synthesized from vapour deposition [6], electrochemical reduction [7], radiolysis reduction [8], thermal decomposition [9], chemical reduction of copper metal salt [10], and room temperature synthesis using hydrazine hydrate and starch [11]. In recent, green synthesis of Cu nanoparticles was achieved by using plant extract [12].

Plant Description

Family Name	: Meliaceae
Binomial name	: *Azadirachta indica*
Common name	: Neem
Plant part taken	: Leaves

Fig. 1 shows the image of neem leaf. Neem is a tree in the mahogany family. Elders find it useful in controlling high blood sugar level and is said to clean up the blood. The tender shoots and flowers of the neem tree are eaten as a vegetable in India. Neem gum is a rich source of protein. Products are believed to be anthelmintic, antifungal, antidiabetic, antibacterial, antiviral, contraceptive and sedative.

Fig. 1 *Azadirachta indica* (Neem)

2. Experimental Methods

2.1 Collection of Sample

Azadirachta indica (neem) leaves were collected within the Sarah Tucker College campus, Tirunelveli during the month of January 2015. Healthy leaves were selected and separated from the rest.

2.2 Preparation of Extract

The selected leaves were washed in running tap water and then again washed with distilled water. Then the leaves were dried with absorbent paper. These leaves were cut into small pieces with a sterile knife. About 25 g of chopped neem leaves was weighed and taken in a beaker and 250 ml of distilled water was added to it. This was heated for 1 hour at 60 °C. By this time aqueous part turns yellow. The extract was filtered by Whatmann No.1 filter paper. This filtrate was made upto 250 mL in a standard measuring flask. It was then stored in refrigerator for further use.

2.3 Reagents and Instruments

Copper acetate ($Cu(CH_3COO)_2 . H_2O$) was obtained from Nice Chemicals, Kochi-682024. Freshly prepared distilled water was used throughout the

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Cite this Article as: S. Ansilin, J. Kavya Nair, C. Aswathy, V. Rama, J. Peter, J. Jeyachynthaya Persis, Green synthesis and characterisation of copper oxide nanoparticles using *Azadirachta indica* (Neem) leaf aqueous extract, J. Nanosci. Tech. 2(5) (2016) 221–223.

experiment. UV-Vis spectrometer (Jasco V-530) and FT-IR (Thermo Scientific Nicolet iS5) were used for characterisation. Surface analysis done by Nanosurf Easy scan 2 AFM.

2.4 Preparation of 0.1 M Copper Acetate Solution

About 4.99 g of copper acetate salt was weighed accurately and made upto 250 mL in a SMF and stored.

2.5 Synthesis of CuO Nanoparticles

For the synthesis of Copper oxide nanoparticles 50 mL of copper acetate and 150 mL of Neem extract is taken and mixed in a beaker. When mixed it is observed that the colour is dark green and brown coloured precipitate is settling down. This obtained precipitate is filtered using Whatmann no-1 filter paper, washed with distilled water and left overnight to dry. The next day CuO nanoparticles were collected (Fig. 2).

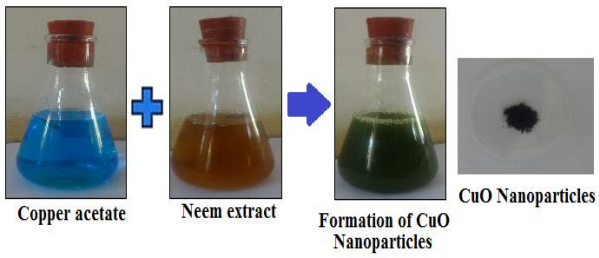

Fig. 2 Synthesis of CuO Nanoparticles

3. Results and Discussion

3.1 UV-Vis Spectra Analysis

UV-Vis spectral analysis of CuO NPs was done in the wavelength range of 200-800 nm. A peak was obtained at 346 nm due to inter band transition of core electrons of CuO NP and the spectrum was represented in Fig. 3. The absorbance wave length values are closely matched with the reported values [13, 14].

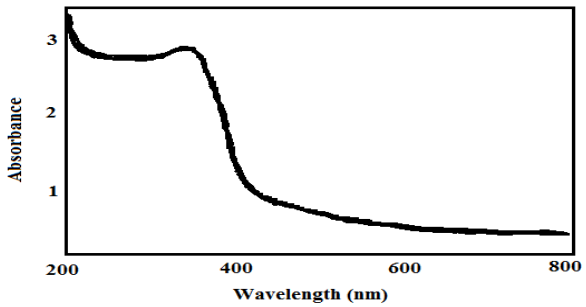

Fig. 3 UV-Vis spectra of CuO nanoparticles

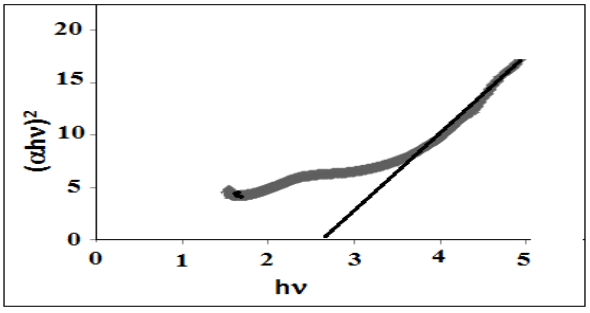

Fig. 4 Tauc plots of CuO nanoparticles

The band gap energy can be determined using the Tauc relation. It is a convenient way of studying the optical absorption spectrum of a material. According to the Tauc relation, the absorption coefficient α for direct band gap material is given by,

$$\alpha h\nu = A(h\nu - Eg)^m$$

where A is the optical constant, α is the absorption coefficient, Eg is the optical band gap and m is an index which assumes the values 1/2, 3/2, 2 and 3 depending on the nature of electronic transition responsible for the reflection.

For determining the band gap energy we used the Tauc plot. Tauc plot has the photon energy (hν) on the X axis and a quantity $(\alpha h\nu)^2$ on the Y axis and extrapolating the linear portion of the curve to the X axis yields the band gap energy of the material. The band gap energy of 2.65 eV obtained for the CuO nanoparticles (Fig. 4).

3.2 FT-IR Measurement

FT-IR graph is taken in the range of 400 to 1500. In Fig. 5, FT-IR spectra peaks at 480.42, 538.80, 811.73, 887.54, 1072.5, 1210.85, 1273.69, 1488.86 cm^{-1}. There is sharp peak observed at 480.42 and 538.80 cm^{-1} in the spectrum of CuO nanoparticles which is the characteristics of Cu-O bond formation. Other peak due to impurity from neem extract.

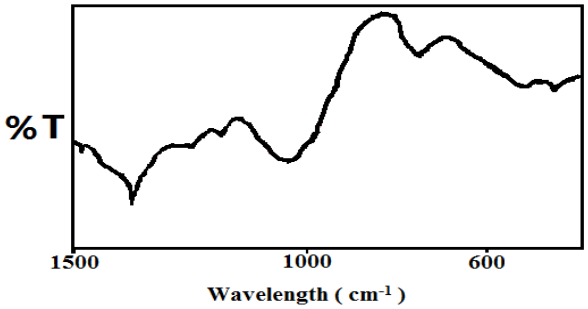

Fig. 5 FT-IR Spectra of CuO Nano particles

3.3 X-Ray Diffraction Studies

XRD pattern of synthesized CuO NPs using a leaf extract of *Azadirachta indica* (Neem) is shown Fig. 6. The XRD pattern shows a high crystallinity of sample level with diffraction angle 2θ of 37.03°, 43.23°, 63.46° and 76.41° which corresponding h,k,l values to the reflection from (111), (200), (220) and (222). The XRD pattern of the CuO Nano particle is crystalline and identical to simple cubic.

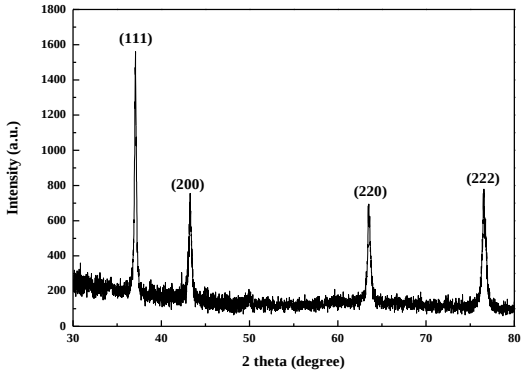

Fig. 6 XRD of CuO Nano particles

3.4 Atomic Force Microscopy

AFM spectra were recorded for CuO nanoparticles. The morphology of the CuO nanoparticles was studied with AFM under the following conditions: a) Scan direction-up b) Time/ Line-206 ms c) Tip voltage-IV d) Vibration frequency-1169.969 KHz e) Measurement environment-air f) Operating mode-Dynamic force. Fig. 7 shows the AFM topographic image of CuO NPs. The particle sizes of CuO NPs are found in different sizes range of 49 nm, 87 nm, 120 nm and 324 nm.

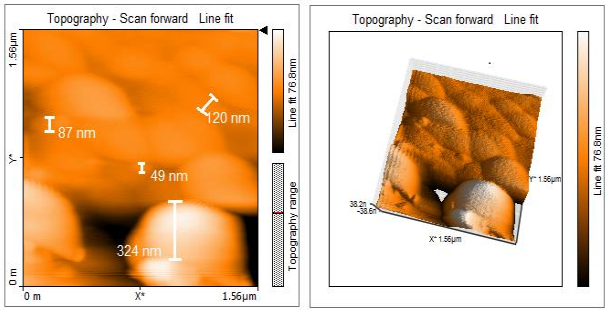

Fig. 7 AFM image of CuO Nano particles

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4. Conclusion

In conclusion, this methods are easily available starting materials, inexpensive and procedure is easy to carry out any laboratory, use of toxic reagent is avoided and pollution free, here we report eco-friendly synthesis of CuO NPs using a leaf extract of *Azadirachta indica* (Neem). The UV-Vis spectra analysis shows that the absorption values for CuO NPs was at 346 nm. In FT-IR spectroscopic analysis there is sharp peak observed at 480.42 and 538.80 cm^{-1} which is the characteristics of Cu-O bond. The XRD analysis confirmed that the obtained NP were crystalline and simple cubic. AFM topography studies of CuO NPs confirmed that the particle size were between 49-324 nm.

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