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Original Research Article

Green synthesis of CuO powder from $CuSO_4$ salt using *Neem* leaf extract and study its characteristics

Priyanka Sangwan[1], Sunita Sharma[2,*], Sonia[1]

[1]Department of Physics, Baba Mastnath University, Rohtak – 124021, Haryana, India
[2]School of Basic Sciences, Galgotias University, Greater Noida – 203207, Uttar Pradesh, India
*Corresponding author, E-mail: sunita.sharma@galgotiasuniversity.edu.in

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ABSTRACT
The present research utilized technology, specifically called green synthesis for extraction of metals and metal oxide nanoparticles (NPs) through physical and chemical methods using *Neem* leaf (Azadirachta India) extract. This eco-friendly method found to be more energy efficient and potentially able to eliminate use of hazardous chemicals compared to other extraction methods. Also, it has much faster reaction rate as compared to other biological or microbial methods. We have synthesized copper oxide (CuO) nanoparticles (NPs) from copper sulphate salt ($CuSO_4.5H_2O$), precursor solution prepared using *Neem* leaf extract. The important compounds in the leaf extract are functional groups as hydroxyl and carbonyl groups which act as reducing agent as well as stabilizing agent. Centrifugation was carried out with the hydrous form of CuO NPs which was derived from *Neem* leaf extract and copper sulphate solution precursor followed by annealing at two different temperatures 450°C and 250°C. The dried sample powder was dark green in color and formed cluster after annealing and drying. The processed powder sample was characterized for structural and morphological property using analytical techniques which comprised of Scanning electron microscopy (SEM), Energy dispersive X-ray Spectroscopy (EDS) and X-ray diffraction analysis (XRD). The SEM images showed granular and agglomerated grains with irregular boundaries that give cluster-like shape. The XRD graph depicted crystallographic planes for monoclinic structure of copper oxide (CuO) NPs along with face-centered cubic structure of copper oxide (Cu_2O) NPs and crystallographic planes of copper (Cu) NPs. The crystalline phase was observed for CuO NPs annealed at 450°C, while some amorphous phase observed for CuO NPs annealed at 25°C but with similar crystalline peaks which were positioned at same 2θ values. This study shows our attempt to synthesize and analyze CuO nanoparticles which have tremendous applications in health, medical and technology sectors without producing toxic chemicals in our environment.

1. Introduction

In the recent years, researchers are showing huge interest in synthesizing metal and metal oxide nanoparticles (NPs) using numerous methods due to their crucial role in nanotechnology. [1, 2] The wide range of application in this diverse domains are cosmetics, pharmaceuticals, healthcare, renewable energy production devices and many more [3]. The nanoparticles have been vastly studied in the last decade for exploring, modifying and utilizing their physical, chemical, mechanical, thermal, opto-electronic and magnetic properties [4]. So far nanoparticles were most of the time synthesized using techniques such as chemical reduction, hydrothermal synthesis, but from the recent decade mostly production carried out using green synthesis methods. The chemical methods can potentially result in toxicity and raise the concerns regarding their effects on human health and environment [3, 5]. Green synthesis refers to the processing techniques which are non-hazardous, sustainable and environmental friendly used for the production of wide range of substances and nanoparticles. These methods use different organisms including fungi, bacteria, algae and parts of plants (seed, fruit, root, stem, leaf etc.); each serving diverse advantages in terms of cost-effectiveness, simplicity and sustainability [6, 7]. Plant extract methodology is a wide term in which parts of plants like seed, stem, root, flower, fruit and leaf extracts are used as reducing agents or catalysts for production of various metals and metal oxides [8-10]. In our research work, we have synthesized nanopartilces of CuO by a cost effective and environmental friendly method using neem (Azadirachta indica) leaf extract used as a reducing agent [11, 12]. Copper and copper oxide NPs synthesized by green methods have grabbed huge attention due to its non-toxicity, cost-effectiveness and modifiable properties in potential applications such as capacitors, gas sensors, solar cell devices and batteries [1, 2, 13]. Conventional chemical methods used to synthesize CuO often accompanied problems such as use of hazardous chemicals and generation of toxic by-products causing damage to the environment [14, 15]. So, leaf extract method is a low cost, highly efficient and harmless process for obtaining metal oxides [7, 9]. The neem extract we have used, provide an excellent medium for the formation of metal oxides due to the oxidative and hydrophilic nature of neem components [11, 12]. The metal oxide formation process includes precipitation, thermal decomposition, sol-gel, spray pyrolysis, electro-deposition and vapor phase decomposition, where the formation of CuO can be found as by product of oxidation of copper metal. The solution (prepared using neem extract and copper sulphate precursor) used as reagent goes under process of thermal decomposition to extract metal oxide. This method was believed to be more efficient and helpful compared to other methods with the formation of the desired product which have potential application in various biomedical problems [3, 13, 14].

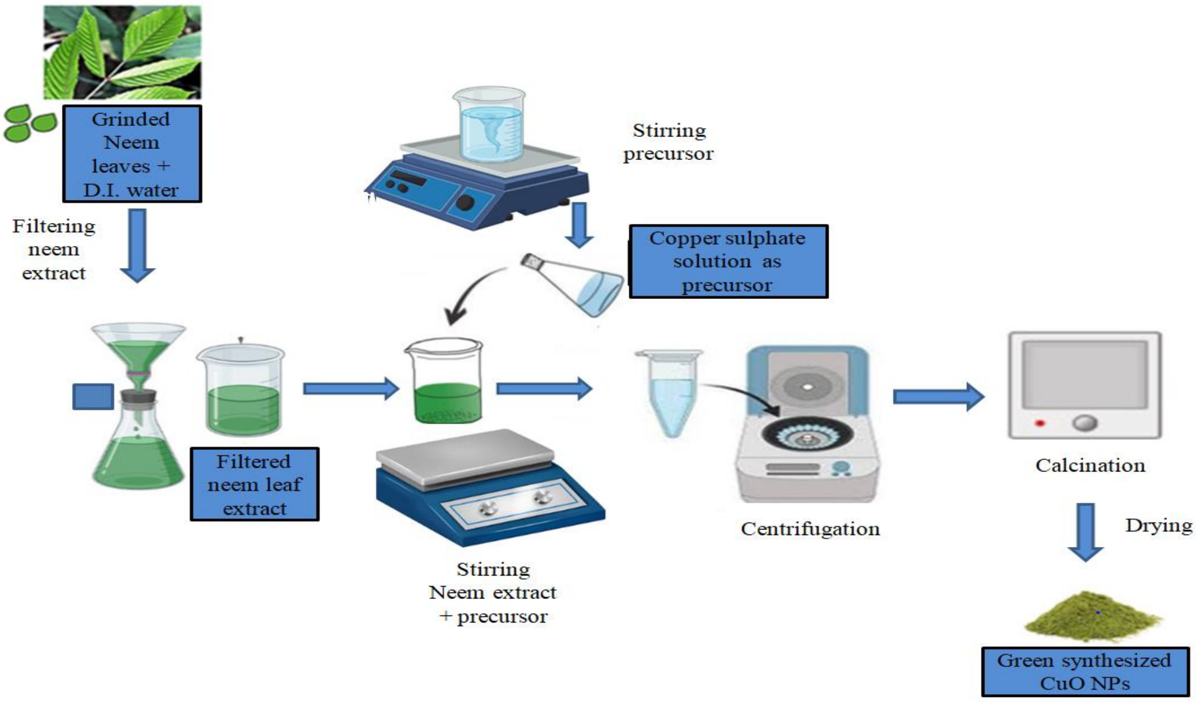


Figure 1: Schematic diagram of Green Synthesis of CuO powder using *Neem* leaf extract.

2. Experimental

Materials required for the green synthesis were easily available and used as fresh. We obtained neem leaves from the campus and copper sulphate salt was available in the laboratory itself.

Synthesis of CuO powder from copper sulphate salt by Green synthesis Method

Material used: Copper sulphate salt ($CuSO_4.5H_2O$), fresh neem leaves, sodium hydroxide (NaOH) pellets, ethanol and D.I. water.

Extraction of neem leave extract: Fresh neem leaves were collected and thoroughly washed several times firstly with tap water to remove dirt and finally with D.I. water 2-3 times to remove any contamination. 200 ml of water was taken along with 50 g of washed neem leaves and mixed in an electric mixer grinder. The thick paste was filtered using a fine sieve and this obtained solution was heated to a temperature of 60°C for 10 minutes while stirring on a magnetic stirrer. It was cooled down to room temperature and left to get settled. Now, the prepared extract of neem was filtered further using Whatman No.1 filter paper twice to get clear solution and removing suspended particles. This obtained neem leaf extract was used as reducing agent for synthesizing CuO NPs [11, 12].

Preparation of precursor solution using $CuSO_4$ salt: To prepare precursor solution of $CuSO_4$ from $CuSO_4$ salt available in the laboratory, we added 20 g of $CuSO_4$ salt in 60 ml of D.I. water taken in a beaker. To mix the solute and solvent we kept the beaker on magnetic stirrer at 60°C for 60 minutes. Solution was kept covered and at R.T. for further use.

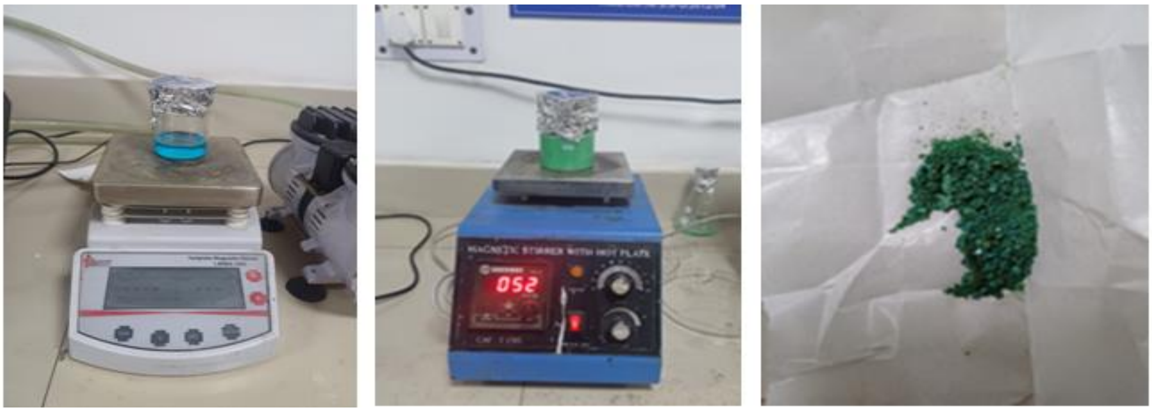


Figure 2: Camera images of (a) Stirring of precursor solution (b) Stirring of precursor and *Neem* extract (c) Annealed (at 250°C) and dried CuO powder.

Green Synthesis of CuO Powder: 80 ml of $CuSO_4$ solution was mixed with 20 ml of filtered neem extract taken in a flask (keeping the ratio 4:1), the pH of this solution was adjusted to 12 by adding two sodium hydroxide (NaOH) pellets. The solution was stirred for 2 hours at 60°C with 3000 rpm. Centrifugation of the solution was carried out in centrifugal machine by firstly adding ethanol and then with D.I. water for washing off the solvent, until we got a clean powder in hydrous form. This thick hydrous paste was collected in china dish and dried at 80°C for 6 hours in hot air oven. Precipitate after completing centrifugation was collected in a ceramic vessel and heated at 400°C for 3 hours to obtain CuO nanoparticles in powder form. Then the residue collected was subjected to crushing for size analysis and further use [9, 11, 16].

Characterization

The structural characteristics and crystalline phases of the samples were examined using a Bruker D8 Advance X-ray diffractometer, utilizing CuKα radiation with a wavelength of 0.15405 nm. XRD measurements were conducted over a 2θ range of 10° to 90° at a scan rate of 3°/min at ambient temperature. The surface morphology of the powder was analyzed using Field emission scanning electron microscopes (FE-SEM), including the TESCAN MIRA II LMH and Emcrafts Genesis-1000 High vacuum models for different resolutions. All the characterizations were performed at room temperature.

3. Results and discussion

Structural Analysis

SEM images, EDX spectra and XRD spectra were analyzed for structural properties.

SEM studies: SEM images in Figure 3(a, b) shows granular and agglomerated grains with irregular boundaries. The formation of dark green viscous liquid which upon heating transformed into solid granules as shown in Figure 2(c) supports the SEM images. Figure 3(a, b) shows SEM images of copper oxide NPs obtained from neem leaf extract and copper sulphate solution precursor at 1K and 30K resolution magnification, respectively of CuO NPs annealed at 450°C and 250°C temperatures. The images show that the CuO NPs were tightly packed together and give cluster-like shape [9,17,18].

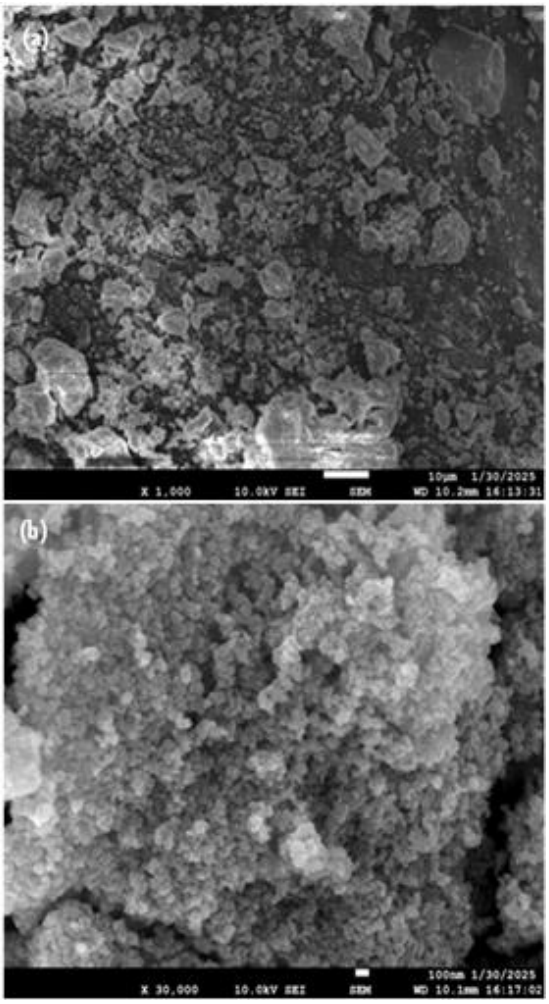


Figure 3: SEM images of (a) CuO NPs at 450°C and (b) CuO NPs at 250°C.

Energy Dispersive X-ray Spectra (EDS) studies: Figure 4 for EDS confirms for elemental compositions of CuO NPs prepared at 250°C. It shows the presence of Cu, O in the obtained CuO NPs.

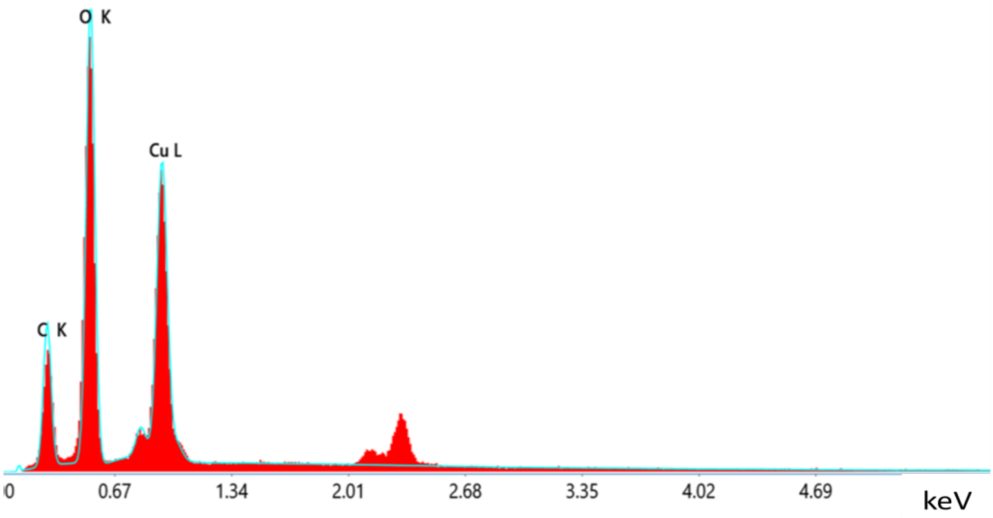
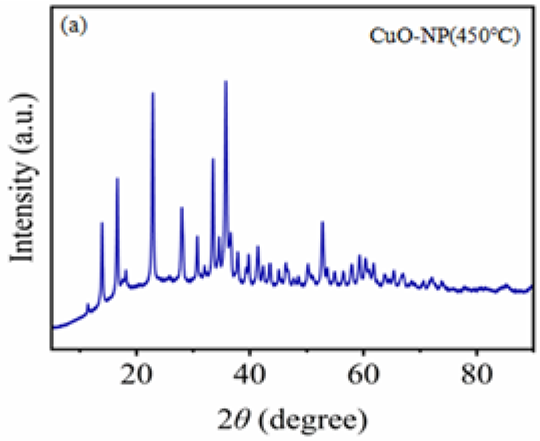


Figure 4: EDS image of CuO NPs.

XRD studies: Figure 5(a, b) shows XRD patterns of CuO powder prepared at temperatures 450°C and 250°C respectively found to be in good agreement with JCPDS files for obtained metal oxide indicating presence of CuO NPs, Cu_2O NPs along with the presence of copper (Cu) NPs. The peaks indexed to crystallographic planes of copper oxide (CuO) NPs with monoclinic structure for (-111), (-202), and (020); face-centered cubic structure of copper oxide (Cu_2O) NPs explicitly for (110), (111), (200), (220), (311), and (222) planes. Also, copper (Cu) NPs were indexed with the diffraction planes (002), (100), and (001). No other diffraction peaks in the XRD spectra signified no impurities present in the obtained powder sample. In Figure 6(a) the crystalline phase observed for CuO NPs synthesized at 450°C, while in Figure 6(b) some amorphous phase observed for CuO NPs synthesized at 250°C, but with almost same crystalline intensity peaks positioned at same 2θ values [19-21].



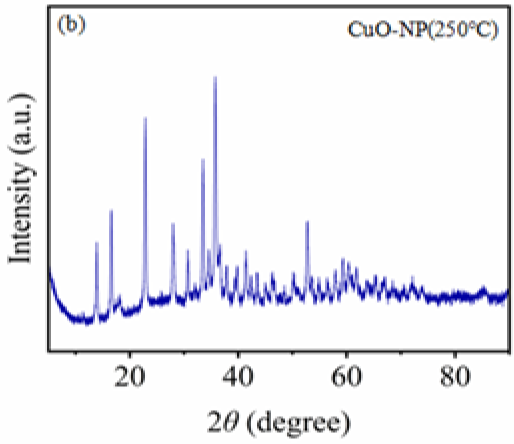


Figure 5: XRD images of graphs of (a) CuO NPs at 450°C and (b) CuO NPs at 250°C.

4. Conclusions

This study gives immense support to the field of green synthesis techniques which uses neem leaf extract as reducing agent and effective catalyst. We successfully synthesized copper and copper oxide nanoparticles by this simple solution based method along with centrifugation process. The precursor material for the synthesis of CuO powder was copper sulphate ($CuSO_4$), an easily available salt which is hazardous free and avoids danger of handling organic solvents. The process was carried out in air and samples were annealed at temperature values of 250°C and 450°C. XRD spectra clearly show that we have obtained not only the bimetallic nanoparticles, but also monometallic nanoparticles. The peaks for crystallographic planes of face-centered cubic structure of copper oxide (Cu_2O) nanoparticles, monoclinic structure of copper oxide (CuO) NPs along with crystallographic planes of copper (Cu) NPs were observed. Possibly due to the presence of a number of bioactive compounds, the reduction of nanoparticles is not complete and forms a mixture of copper and copper oxide nanoparticles. The crystalline phase was observed for CuO NPs annealed at 450°C while, some amorphous phase observed for CuO NPs annealed at 250°C, but with same crystalline peaks positioned at same 2θ values. Green synthesis methods have restricted control over the size and shape of the resulting nanoparticles that can have an impact on the properties of nanoparticles and their applications. But our green synthesized copper and copper oxide nanoparticles have good stability and longer shelf life for varying temperatures as compared with nanoparticles obtained with any conventional chemical method. This stability provides capability for storage and practical efficacy of the produced metallic oxide nanoparticles. Thus, considering the foremost concern nowadays in research of developing sustainable methodologies for environmental protection; leaf extract method proved to be a good option for using neem extract as a reducing agent in the synthesis of metal or metal oxide NPs.

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Authors' contributions

Priyanka Sangwan has done all the sample preparation related experimental work, data analysis and paper writing. Dr. Sunita Sharma did the proof reading of the research paper. Dr. Sonia did reading of the manuscript. The author read and approved the final manuscript.

Conflicts of interest

The author declares no conflict of interest.

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Data availability

No new data were created.

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