

Research article

Characteristics of Synthesized Copper Oxide (CuO) Nanoparticles using Maize Husks: A Green Chemistry Approach

Abiola Olawale Ilori, Olanrewaju Ajanaku

Department of Physical Sciences, Olusegun Agagu University of Science & Technology, Okitipupa, Ondo State, Nigeria.

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Abstract: Nanoparticles of a transitional metal oxide such as Copper (II) oxide or cupric oxide have gained considerable interest in the research and development community due to the corresponding conventional materials' extremely different characteristics. Hence, this study aimed at characterizing Copper (II) oxide nanoparticles synthesized through a direct green chemical approach using the outer leafy cover of the maize husk. The fresh maize husk's optical absorption spectrum shows broad peaks centered around 209, 241, and 331 nm, which are the characteristic of flavonoids. The structural analysis shows the formation of Nantokite with a face-centered crystal structure, which has crystalline peaks (111), (220), (311), and (331) at $2\theta = 28.5, 47.4, 56.28, \text{ and } 76.6$, respectively. In contrast, the spectrum of Copper (II) oxide showed a base-center monoclinic crystal structure with lattice parameters: $a = 4.688, b = 3.4229, c = 5.1319$ and $\beta = 99.91$. In conclusion, nanoparticles' synthesis using aqueous corn husk extract was successfully synthesized and characterized. This environmentally friendly approach is a simple, environmentally friendly, inexpensive process and is reproducible.

Keywords: Characterization; Nanoparticles; Synthesis; UV-VIS; X-Ray Diffraction (XRD).

1. Introduction

The research on synthesis and characterization of metallic nanoparticles is an emerging field of nanotechnology due to the applications of nanoparticles for scientific, technological, pharmacological, and biomedical sectors. Various physical and chemical routes have been employed to synthesize nanoparticles [1]. The chemical method of synthesizing nanoparticles is a highly costly affair with additional environmental and biological risks. However, biogenic synthesis, which involves plants to produce nanoparticles, is a cheap, environment-friendly, and less expensive method [2], [3].

Nanoparticles of transitional metal oxide have gained considerable interest in the research and development

community due to the highly different characteristics of these nanoparticles compared to the corresponding conventional materials [4]–[6]. High-quality, well-defined, and configurable nanoparticles are crucial prerequisites for developing nano-devices and their applications [4], [7]–[9].

Cupric oxide, CuO, which is a p-type semiconductor [10], (indirect bandgap of 1.2 to 1.5 eV), and a transition metal-oxide, has been widely exploited for diverse applications [11]. It has a monoclinic configuration with one atom of copper linked to four oxygen atoms [12]. CuO is blackish and has many unique properties, including hyperthermal conductivity, distinctive electrical and magnetic properties, high absorption coefficient, abundance in nature, photovoltaic properties, high stability, and antimicrobial activity [4], [13].

Copper Oxide (CuO) can be used for several technological applications such as catalytic materials [8], gas sensors [14], [15], high-efficiency thermal conductive materials [16], reliable selective magnetic media, or solar cells [5]. Many synthetic methods contribute an essential part of the fundamental study to understanding properties and discovering applications based on nanoparticles. It enables scientists to monitor different parameters for the nanoparticles, such as morphology, particle size, distribution, and composition. It is essential to prepare CuO nanoparticles through a simple, cost-effective, and eco-friendly process that can yield uniform nanoparticles in sizes and shapes.

Copper Oxide (CuO) nanoparticles have been prepared via various methods such as thermal evaporation, nonchemical, sol-gel hydrothermal, electrochemical, and microwave irradiation method [17]–[22].

This study aims to characterize the structure of the CuO nanoparticles synthesized through a direct green chemical approach using plant extracts to bio-oxidize or bio-reduce metal salts and examine their optical properties. This method offers advantages over physical processes because it is cost-effective, reproducible, environmentally friendly, and efficient.

2. Material and Methods

This study's sample of interest is the corn or maize husk's outer leafy cover known as *Zea mays*. Maize husk derivatives produced through phytochemical experiments consist of flavonoids, saponins, alkaloids, and glycosides, while tannins and phenols are in the methanol derivatives [23], [24]. Natural extracts from the fresh maize husk have been used to synthesize copper oxide nanoparticles, and analyses were carried out on their optical absorption spectrum and structural properties.

2.1. Preparation of the Materials

Fresh maize husks weighing 4.6 gr were chopped into small pieces and placed in a beaker containing a magnetic stirrer and 200 ml of deionized water. The beaker's content was then heated using a hot plate to 70°C and held at that temperature for about 50 minutes, yielding a light yellowish transparent extract. The extract was cooled to room temperature and periodically filtered with a paper filter (Whatman No. 1). 1.5 gr of copper chloride (CuCl) manufactured by Lab-Aids Inc. with 10% purity was introduced to 100 ml of the extract and stirred at room temperature for 17 hours leading to complete dissolution of the CuCl in the extract [25]–[27].

Consequently, the color of the resulting solution changed from transparent light yellowish to light green. 50 ml of the resulting solution was oven-dried at 100°C for 7

hours. In the end, a green powder was obtained. The remaining 50 ml of the resulting solution was taken for UV-VIS analysis. The green powder was annealed in a furnace at 600°C for 2 hours resulting in black powder. Figure 1 shows the process of synthesizing nanoparticles.

2.2. Instrumentation Experimental Procedure

The optical measurements were performed with a double beam Cary 5000 UV-VIS-NIR spectroscope. In contrast, the phase characterization was performed using the Bruker XRD spectrometer using Cu-K α ($\lambda = 1.54 \text{ \AA}$, voltage = 40kV, current = 40 mA, and 2θ between 00 to 1000).

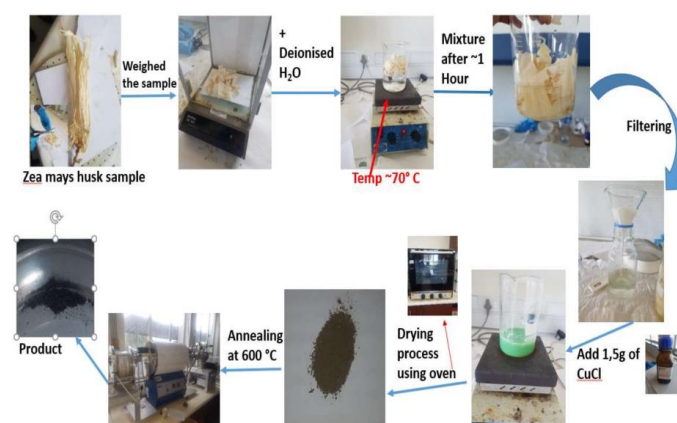


Figure 1. CuO Nanoparticles Synthesized Processes.

3. Result and Discussions

3.1. The Optical Absorption Spectrum

Broad peaks centered around 209, 241, and 331 nm were observed. These peaks are characteristic of flavonoids. The results conform with the study [28], [29], which showed that flavonoids spectra typically consist of two absorption peaks in the ranges 230–285 nm (a band I) and 300–380 nm (band II). Figure 2 displays the optical absorption spectrum for fresh maize (*Zea mays*) husk extract. The figure shows that the absorbance of the extract transit towards the positive wavelength for the first two absorption peaks, which conforms to the results [26].

From the results, Band I and Band II conform to absorption by the A-ring benzoyl and the B-ring cinnamoyl systems, respectively [30]. As shown in Figure 2, the absorbance of the extracts increased towards the positive wavelength, which confirms that fresh extracts at lower temperatures are highly efficient for the bioactive components. As seen from the results, the broad absorption peak indicated a high reflectance property within the infrared region, which is much more due to already existing organic plant extracts in the sample. The reflectance decreased significantly after annealing, and the

peaks of absorption in the infrared region disappeared, suggesting that the organic plant extracts had burnt off.

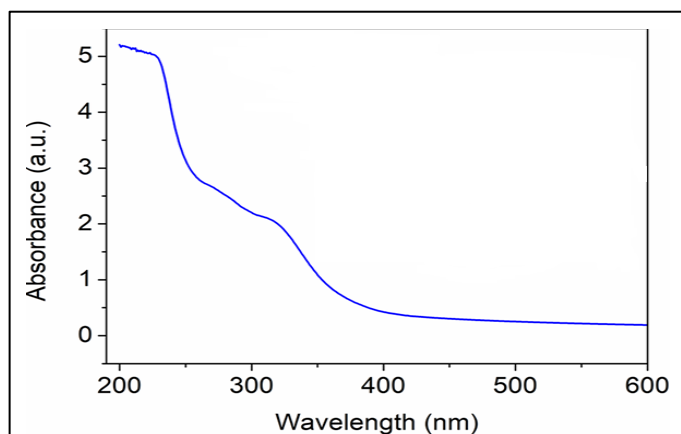


Figure 2. UV-VIS Characterization (Optical Absorbance Spectrum) of CuO Extract from Fresh Maize Husk.

3.2. Structural Analysis

X-Ray Diffraction (XRD) analysis of the green powder (black line) showed CuCl nanoparticles' formation, also known as Nantokite, with a face-centered (space group of F-43m) crystal structure. The crystalline peaks (111), (220), (311) and (331) at $2\theta = 28.5, 47.4, 56.28,$ and $76.6,$ respectively which resembles the reference diffractogram of FCC CuCl pattern #00-006-0344 with lattice parameter: $a = 5.4160$. Upon annealing CuCl at a temperature $\sim 600^\circ\text{C}$ for 2 hrs, CuCl was completely transformed to pure CuO (red line).

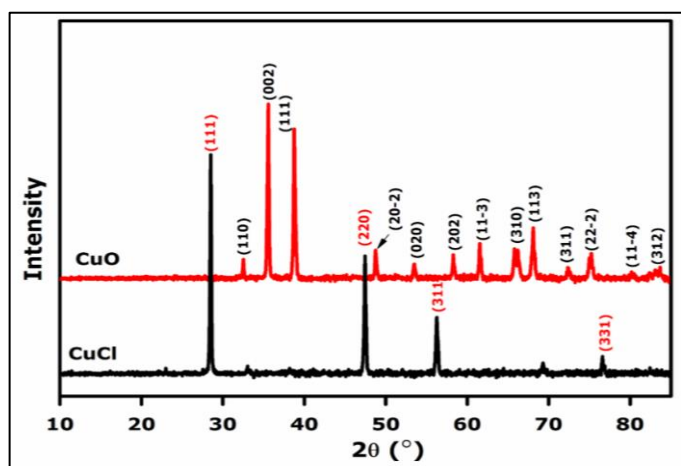


Figure 3. XRD Spectrum for The Dried Husk Extracts Powder (Unannealed) and The Annealed Sample.

The XRD spectrum of CuO showed a base-center monoclinic (space group C2/c) crystal structure resembling the reference diffractogram pattern #00-048-1548 with lattice parameters: $a = 4.688, b = 3.4229, c = 5.1319$ and $\beta = 99.91$.

The characteristic observed at $2\theta = 32.5, 35.5, 38.73, 48.76, 53.5, 58.2, 61.5, 65.7, 66.24, 68.07, 75.2, 80.2,$ and 83.1 are assigned to the (110), (002), (111), (20-2), (020), (202), (11-3), (310), (113), respectively, as shown in Figure 3.

As logarithmic law specifies, the activation energy for Cu₂O oxidation is relatively high below 800°C [31]. Still, it becomes significantly low or negative at temperatures above $800-1050^\circ\text{C}$, which could be accomplished by illuminating the sample with a high laser intensity beam [32].

For this study, the phytochemicals present in the copper oxide synthesis extract may have induced the cooling effect needed for the complete thermal oxidation of Cu₂O to CuO. This temperature reduction leads to creating a pure CuO at 600°C . The drop in crystal size with annealing temperature, on the other hand, is due to phase transitions in crystal lattice and deformations.

4. Conclusion

The synthesis of CuO nanoparticles using aqueous corn husk extract was successful, and the optical properties were studied. The resulting nanoparticle resembles the structural properties of known materials CuCl and CuO. The transparent light green Cu₂O nanoparticles thermally oxidized to pure monoclinic CuO nanoparticles were formed at 600°C . In the visible region, the annealed samples exhibit high absorption properties. Consequently, it can be used to develop nano-devices for various technological applications. This environmentally friendly approach is a simple, inexpensive process and is reproducible.

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