NanoNEXT

Short Communication

DOI: 10.54392/nnxt2321

Facile Synthesis and Characterization of Spindle like Copper Oxide Nanostructures

K. Girija [a,*]

[a] Department of Physics, Dr. N.G.P. Arts and Science College, Coimbatore-641048, Tamil Nadu, India
*Corresponding author Email: girija.k@drngpasc.ac.in
DOI: https://doi.org/10.54392/nnxt2321
Received: 29-04-2023; Revised: 13-06-2023; Accepted: 20-06-2023; Published: 27-06-2023

Abstract: The study is dedicated to a comprehensive examination of copper oxide nanoparticles, with a primary focus on their fabrication and systematic analysis. The principal objectives encompass the synthesis of CuO nanoparticles through a straightforward precipitation method, the structural characterization through X-ray diffraction (XRD), the determination of particle size and morphology using transmission electron microscopy (TEM), the analysis of functional groups within the CuO nanoparticles via Fourier transform infrared (FT-IR) spectroscopy, and the exploration of absorbance peaks utilizing UV-Vis spectroscopy. The research outcomes unlock novel prospects for scientific and technological advancements, offering crucial insights into the intrinsic properties and potential applications of copper oxide nanoparticles.

Keywords: Nanoparticles, XRD, TEM, FT-IR

1. Introduction

Block d and block p elements from the periodic table, namely copper and oxygen, intricately bond to create copper oxide, with four oxygen ions coordinating around each copper ion within the crystal lattice. Given copper's vital role in modern technology and its widespread availability, there is a significant focus on copper (Cu) and copper oxide (Cu2O) nanoparticles [1]. The escalating interest is due to the diverse range of optical, catalytic, mechanical, and electrical properties exhibited by copper nanoparticles [2, 3]. Copper (Cu) is a vital trace element essential for the well-being of humans, plants, and animals [4], [5]. Despite its minimal requirement in humans [6], its significance cannot be understated. An average adult weighing approximately 70 kg contains around 100 mg of copper within their system [7, 8]. The daily intake of copper should ideally range from 2-4 mg, with an upper limit of 10 mg, predominantly sourced from dietary consumption through food and beverages. Copper plays a multifaceted role in human physiology, participating in various processes. These encompass the synthesis of neuropeptides, regulation of cell signaling pathways, bolstering antioxidant defense mechanisms, and facilitating the immune cells' function in pathogen elimination [8]. Copper nanoparticles (Cu-NPs) find versatile applications in coatings, plastics, and textiles, serving as effective agents with antibacterial, antioxidant, antidiabetic, anti-inflammatory, and antifouling properties [9, 10]. Moreover, their utility extends to domains such as anticancer treatments, heat transfer fluids, sensors, lithium-ion batteries, gas sensors, e-sensitized solar cells, and heterogeneous catalysis [11, 12]. The aim of this study was to investigate structural and optical properties of copper oxide.

2. Experimental Procedure

Nanoparticle production typically involves a series of steps, including preparing the precursor solution, incorporating additives or surfactants for nanoparticle modification, followed by heat treatment, washing, and drying processes. Similarly, in this study, a procedure was followed where 50 ml of double-distilled water was poured over 2g of copper acetate, and stirring was carried out for 20 minutes. Gradually, NaOH solution was added drop by drop until the pH reached 10. Subsequently, a solution of triethanolamine acetate (3 g) was introduced after 1 hour of stirring and maintained at a continuous heat of 80 °C for 3 hours. The resulting solution underwent centrifugation at 1200 rpm using distilled water.

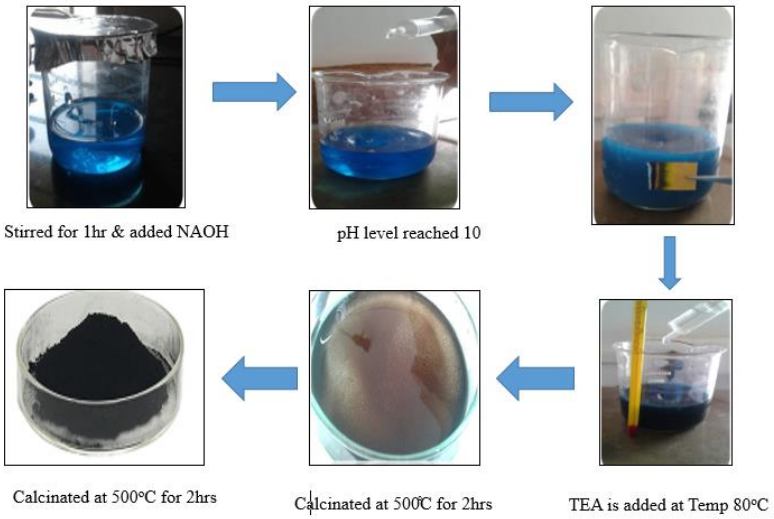

Figure 1. Schematic Illustration of the procedure

It was then dried on a hotplate at 60 °C for 45 minutes, followed by calcination in a muffle furnace at 500 °C for two hours, leading to the formation of CuONanoparticles. Figure 1 shows schematic Illustration of the procedure.

3. Results and Discussion

3.1 Structural Analysis

Aqueous precipitation was employed for the synthesis of copper oxide (CuO) nanoparticles, utilizing copper acetate as a precursor and NaOH as a stabilizing agent to maintain a pH level of 10. This technique facilitates the simplified large-scale production of CuO nanoparticles. The structural characteristics of the resulting samples, including phase, purity, and crystallinity, were evaluated through X-ray diffraction (XRD) analysis. The Figure 2. shows that XRD pattern of nanoparticle. Investigation via X-ray diffraction confirmed the presence of a monoclinic crystal phase in the CuO material, which aligns consistently with JCPDS Card No. (45-0937) for all identified diffraction peaks.

The monoclinic crystal structure of CuO was discerned through X-ray diffraction analysis, with distinctive peaks observed at 2θ angles of 32.41°, 35.39°, 38.81°, 48.47°, 61.54°, 66.09°, 68.08°, and 74.90°. These angles correspond to the crystallographic planes (-110), (111), (022), (-202), (202), (-113), (022), (-220), and (-222), respectively. It was found that the lattice parameters were a = 0.4685 nm, b = 0.3425 nm, and c = 0.5130 nm. The presence of a distinct and condensed diffraction peak at 35°, which corresponds to the (111) plane, shows that the samples have a high degree of crystallinity. In the ready samples, there were no peaks associated with impurities.

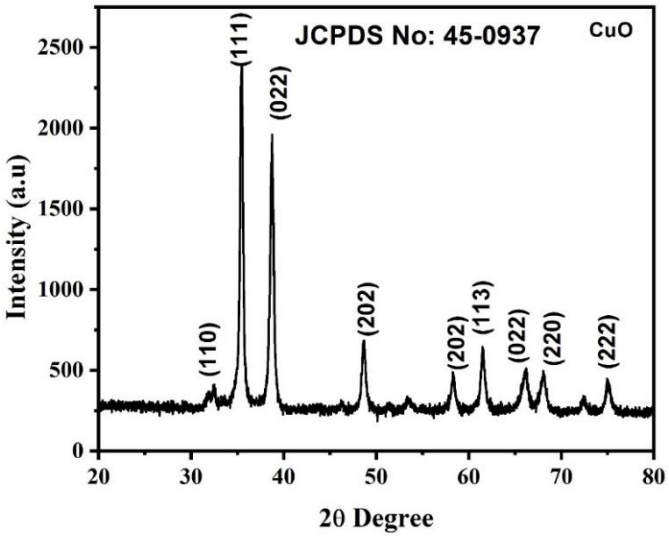

Figure 2. XRD pattern for CuO nanoparticles

The crystallite size of the CuO is obtained by Debye scherrer formula,

$$D = k\lambda/\beta \cos\theta$$

where, D - Size of the crystal in the direction perpendicular to the reflecting plane

k - Proportionality constant = 0.94

λ - Wavelength of x-ray = 1.54 Å

β - The Full Width at Half Maximum of the peak in radian

θ - Diffraction angle

CuO nanoparticles were synthesized using the Debye-Scherrer formula, revealing an average crystallite size of 14 nm.

3.2 Shape and Particle Size Analysis

Transmission Electron Microscope (TEM) measurements were conducted to elucidate the morphology of Copper Oxide Nanoparticles, as depicted in Figure 3 (a) and (b). Notably, the CuO material exhibits spindle-like nanostructures, discernible in the low-magnification TEM image presented in Figure 3 (a). Upon closer inspection in the magnified TEM image of Figure 3 (b), it becomes evident that the nanosphere-like particles intricately self-assemble, forming the distinctive nanostructured CuO material. The CuO nanoparticles manifest a relatively uniform diameter of approximately 20 nm.

Consequently, the crystallite size derived from XRD analysis and the observed particle size of CuO nanoparticles from TEM examination closely correspond, reinforcing the congruence between the two analytical approaches.

3.3 Fourier Transform - Infra Red (FT-IR) Analysis

Utilizing a Thermo Nicolet V-200 FT-IR Spectrometer and the KBr pellet technique, the FT-IR spectroscopic analysis of CuO nanoparticles was conducted across the mid-infrared spectrum, ranging from 4000 cm^{-1} to 400 cm^{-1}. The functional groups within the synthesized sample were discerned by analyzing the FT-IR spectra of CuO nanoparticles, which are illustrated in Figure 4. The absorption peak at 3717 cm^{-1} in the spectra may be attributed to O-H stretching vibrations coupled with copper atoms, indicating a potential interaction.

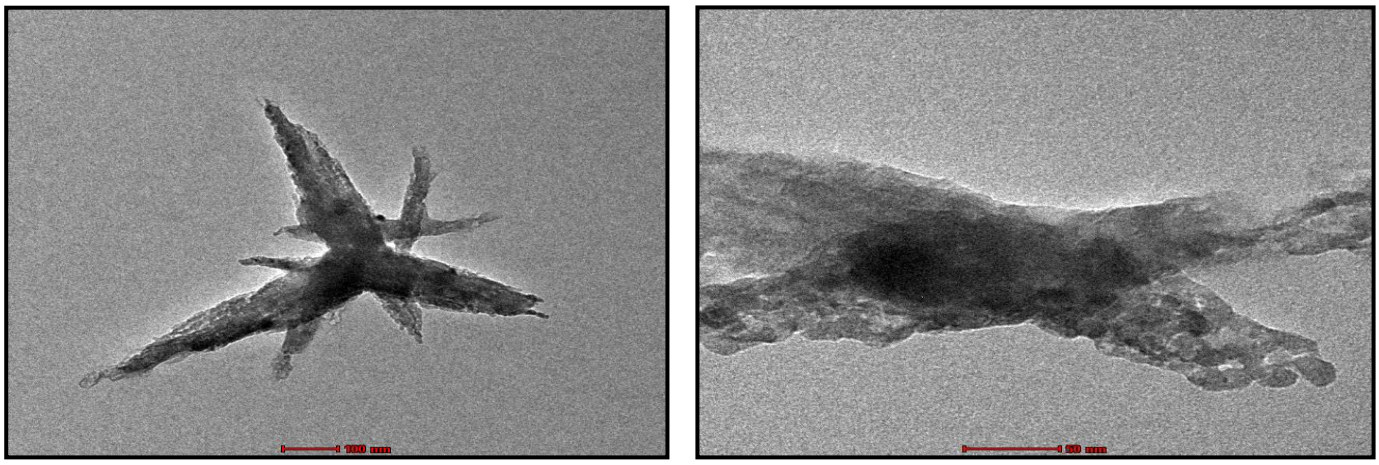

Figure 3 (a) Transmission electron microscope image of CuO, **(b)** Magnified

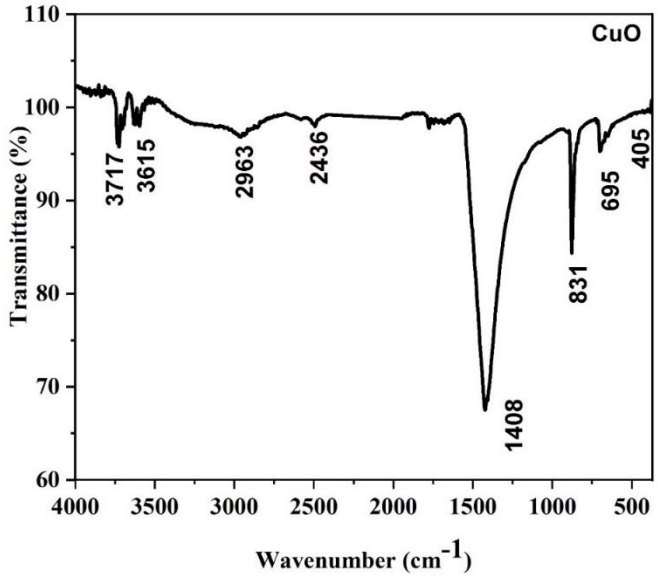

Figure 4. FTIR spectrum of CuO nanoparticle

Likewise, the presence of adsorbed water molecules due to the high surface-to-volume ratio of nanocrystalline materials could lead to the peaks at 3615 cm^{-1}. A broad absorption peak observed at approximately 2963 cm^{-1} is likely linked to the presence of ambient CO_2. Additionally, a spectrum with low intensity at 2436 cm^{-1} may indicate the existence of an organic molecule associated with the -CH_2 group.

The presence of carbonyl C=O stretching bonds is evident in the pronounced and intense bands observed at 1408 cm^{-1}. Additionally, the spectrum displays subtle vibrational bands centered at 831 cm^{-1}, representing the asymmetric stretching of -COOH groups. The formation of Cu-O bonds is highlighted by a prominent peak at 695 cm^{-1}, while the distinct stretching vibration of the Cu-O bond in CuO leads to the peak at 405 cm^{-1}. Notably, every discernible peak in the spectrum corroborates the successful production of CuO nanoparticles.

3.4 UV-Visible Spectrum Analysis

The optical characteristics of the prepared materials can be efficiently explored using the UV-visible absorption spectroscopy technique. This approach allows for a straightforward investigation of electronic transitions and energy band properties through absorption spectra.

Following the dissolution of the CuO nanostructure in distilled water, UV-Vis measurements were conducted. By locating the absorption edge, the bandgap value and type of transition were determined. The absorption spectra were employed to analyze the type of electronic transitions and energy band behavior. As depicted in Figure 5., the absorption spectra of CuO nanoparticles reveal a fundamental absorption edge occurring around 340 nm, attributed to a direct transition of electrons. This phenomenon is likely attributed to the absorption of light resulting from the stimulation of electrons from the valence band to the conduction band within the CuO nanostructures. A similar observation was reported in previous studies, where an absorption band at a wavelength of 345 nm was identified [13]. The absence of additional peaks in the spectrum serves to confirm the pure nature of the synthesized sample as CuO. Through the employment of the formula $Eg = 1240/\lambda$, the optical band gap can be deduced based on the absorbance edge in the CuO nanoparticles' absorbance spectrum. The calculation yielded a direct band gap value of 3.6 eV. This significant finding suggests that the CuO nanoparticles produced via wet chemical synthesis hold promise for applications in sensing, photocatalysis, and photovoltaics.

4. Conclusion

This paper presents a comprehensive overview of the facile precipitation method employed for the synthesis of CuO nanoparticles. A range of techniques has been employed to thoroughly characterize the resulting copper oxide nanoparticles, encompassing investigations into their structural attributes, morphology, size, functional groups, and band gap energy. X-ray Diffraction (XRD) analyses confirmed the presence of monoclinic phase copper oxide nanoparticles synthesized at a pH of 10. The crystallite size was ascertained to be 14 nm. Transmission Electron Microscopy (TEM) further validated the size and morphology of the CuO nanoparticles, revealing spindle-like nanostructures formed through the self-assembly of sphere-shaped nanoparticles. A particle size of approximately 20 nm was determined, which aligns well with the findings from the XRD study. Functional groups are identified through Fourier Transform Infrared (FTIR) spectroscopy, where peaks corresponding to O-H vibration bonds are prominent at 3717 cm^{-1}. The formation of Cu-O bonds is evident from a robust peak observed at 695 cm^{-1}. The distinctive stretching vibration of the Cu-O bond within CuO is characterized by the peak at 405 cm^{-1}. The determination of band gap energy was achieved through the analysis of absorption spectra. The absorption spectra of CuO nanoparticles exhibit a fundamental absorption edge around 340 nm, which can be attributed to the direct transition of electrons. By calculating the optical band gap based on the absorbance spectra, it was determined that the band gap of CuO nanoparticles is 3.6 eV.

Reference

[1] M.J. Guajardo-Pachecoa, J.E. Morales-Sanchz, J. González-Hernándezc, F. Ruiz, Synthesis of copper nanoparticles using soybeans as a chelant agent. Materials Letters, 64 (2010) 1361-1364. [DOI]

[2] Y. Xi, C. Hu, P. Gao, R. Yang, X. He, X. Wang, B. Wan, Morphology and phase selective synthesis of Cux O (x = 1, 2) nanostructures and their catalytic degradation activity. Materials Science and Engineering: B 166 (2010) 113-117. [DOI]

[3] Y. He, A novel solid-stabilized emulsion approach to CuO nanostructures microspheres.

Materials Research Bulletin, 42 (2007) 190-195. [DOI]
[4] M.C. Linder, M. Hazegh-Azam, Copper biochemistry and molecular biology. The American journal of clinical nutrition. 63 (1996) 797-811.
[5] S. Raha, R. Mallick, S. Basak, A.K. Duttaroy Is copper beneficial for COVID-19 patients?. Medical Hypotheses, 142 (2020) 109814. [DOI]
[6] M. Bost, S. Houdart, M. Oberli, E. Kalonji, J.F. Huneau, I. Margaritis, Dietary copper and human health: Current evidence and unresolved issues. Journal of Trace Elements in Medicine and Biology, 35 (2016) 107-115. [DOI]
[7] Z. Shabbir, A. Sardar, A. Shabbir, G. Abbas, S. Shamshad, S. Khalid, N. Natasha, G. Murtaza, C. Dumat, M. Shahid, Copper uptake, essentiality, toxicity, detoxification and risk assessment in soil-plant environment. Chemosphere. 19 (2020) 127436. [DOI]
[8] M. Latorre, R. Troncoso, R. Uauy. Biological Aspects of Copper. In Clinical and Translational Perspectives on Wilson Disease, (2019) 25-31. [DOI]
[9] A.T. Apostolov, I.N. Apostolova, J.M. Wesselinowa, Dielectric constant of multiferroic pure and doped CuO nanoparticles. Solid State Commun. 192 (2014) 71–74. [DOI]
[10] M. Thiruvengadam, I.M. Chung, T. Gomathi, M.A. Ansari, V.G. Khanna, V. Babu, G. Rajakumar. Synthesis, characterization and pharmacological potential of green synthesized copper nanoparticles. Bioprocess Biosyst Eng. 42 (2019) 1769–1777. [DOI]
[11] O. Rubilar, M. Rai, G. Tortella, M.C. Diez, A.B. Seabra, N. Duran, Biogenic nanoparticles: copper, copper oxides, copper sulphides, complex copper nanostructures and their applications. Biotechnol Lett. 35 (2013) 1365–1375. [DOI]
[12] O. Waser, M. Hess, A. Guntner, P. Novak, S.E. Pratsinis, Size controlled CuO nanoparticles for Li-ion batteries. Journal of Power Sources, 241 (2013) 415-422. [DOI]
[13] H.R. Ghorbani, I. Fazeli, A.A. Fallahi, (2015). Biosynthesis of copper oxide nanoparticles using extract of E. coli. Orient Journal of Chemistry, 31(1), 515-517. [DOI]

Does this article screened for similarity?

Yes

Conflict of interest

The Author declares that there is no conflict of interest anywhere.

About the License