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Original Research

Studies of Structural and Optical Properties of Copper oxide (CuO) Nanoparticles

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Abstract

Nanoparticles of copper oxide (CuO) synthesized by the co-precipitation method with copper (II) chloride dihydrate (CuCl2.2H2O) as a precursor and sodium hydroxide (NaOH) as a stabilizing agent are the subject of this investigation into their optical and structural characteristics. A 5-hour sintering process was used on the prepared sample at 600 oC. The synthesized sample exhibited a single-phase monoclinic structure with an average crystal size of 12.85 nm, as revealed by the XRD result. Micrographs taken using scanning electron microscopy (SEM) revealed that the sample's grains were uniformly agglomerated and not round. The UV-vis spectroscopy method was used to measure the optical properties. The absorption peak was determined to be at around 272 nm, which corresponds to an optical band gap energy of 3.59 eV, according to the UV-vis measurements. A few examples of CuO's numerous possible uses include photocatalysts, solar energy converters, sensing devices, supercapacitors, lithium-ion battery electrodes, and photothermal and photoconductive uses.

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INTRODUCTION

Although nature is rich with metal oxides, the relative usefulness of these compounds is highly dependent on their applications in contemporary technology and science (Luna, 2015). The periodic table is populated with transition metals, which serve a variety of purposes across many different industries (Lanje, 2010). A notable group of semiconductors known as transition metal oxides have applications in fields such as electronics (Medhi, 2019), catalysis (Haider, 2020), magnetic storage (Priyadharsini, 2020), and solar energy conversion (Singh, 2016). Sagadevan (2017) noted that some transition metal oxides, such as ZnO, TiO_2, NiO, and Fe_3O_4, have shown promise in several fields. Unlike their bulk metallic counterparts, CuO nanoparticles exhibit semiconductor behavior at the nanoscale, making them unique. Like other oxides of precious metals, CuO finds extensive usage in many different types of manufacturing. Photothermal and photoconductive uses make use of CuO, a tiny band-gap semiconducting material (Koshy,

2011). CuO's huge band gap is nearly identical to ZnO's, making it very useful in the nanorange and for making supercapacitors (Nagarani, 2022). An ideal material for solar cell windows and energy conversion, CuO has a band gap of 2.6 eV (Lanje, 2010). Copper oxide nanoparticles are intriguing because of how well they work as nanofluids to cool refrigerators. Sonochemical procedures with sizes of 20-30 nm have been reported as one of several recent ways for the preparation of CuO nanocrystalline (Salavati-Niasari, 2009). According to Aparna (2012), sol-gel produced CuO nanoparticles with a size ranging from 1 to 10 nm. According to Patel (2017), CuO nanoparticles with an average size between fifteen and twenty nanometers were synthesized utilizing the solid-state reaction approach at ambient temperature. According to Barman (2017), CuO nanoparticles measuring around 4 nm were synthesized electrochemically. In this study, we use a straightforward co-precipitation technique to create 12.854 nm CuO nanoparticles.

MATERIALS AND METHODS

Experimental

The precursors utilized to fabricate CuO nanoparticles were solutions of copper (II) chloride dihydrate with stoichiometric weights ($CuC_{l2}.2H_2O$). The reaction mixture included ethanol as the reactive agent, sodium hydroxide (NaOH) as the stabiliser, and likely deionized water as the solvent. The solution was agitated for 30 minutes after adding 12.0 g of $CuC_{l2}.2H_2O$ to 100 mL of DI water to create a 0.7M solution. Sodium hydroxide (NaOH) weighing 9 grams was dissolved in 150 milliliters of DI water in its own right. A solution of sodium hydroxide was slowly added to a solution of copper (II) chloride dihydrate while being constantly stirred at 70 ˚C until a change in color was seen. After two hours at 0˚ C, the solution's color changed from green to bluish green and then to a black precipitate as the reaction continued. Copper hydroxide, a dark precipitate, was passed through a filter paper. After that, the sodium chloride salt solution was removed by washing it four or five times with a mixture of deionized water and ethanol. Thereafter, the precipitate was subjected to hot air drying for 8 hours at a temperature of approximately 100 ˚C. In a temperature-controlled electric furnace, the dry sample was sintered for 5 hours at 600˚C to produce crystalline CuO-NPs. To obtain crystal nanoparticles, the sintered material was further coarsely ground many times using agate mortar. A scanning electron microscope (SEM) and an X-ray diffractometer (XRD) were used to characterize the structure and morphology of the powder after it had been finely ground. The UV-Vis spectroscopy method was used to measure optical parameters.

Characterization Techniques

The structural and phase analyses were defined with the use of a CuKα radiation source (λ = 1.5405 Å), an XRD system from Philips Holland (PW 1710), a step size of 0.020, and a scan rate of 4 degrees per minute. A JEOL JSM 5600 scanning electron microscope was used to examine the copper oxide nanoparticles' morphology. A scanning electron microscope (JSM-7610F) was used to

analyze the surface morphology of the prepared sample. This type of microscope generates signals from the sample by focusing a powerful electron beam. By enlarging the shown patterns to magnifications between 500x and 5,000kx, these signals provide details regarding the sample's exterior morphology. A spectrophotometer model SHIMADZU UV-1800 was used to acquire the absorption spectra in the ultraviolet and visible light.

RESULTS AND DISCUSSION

Structural study

The XRD pattern of CuO nanoparticles is displayed in Figure 1 (Hameed, 2022). Crystalline material with a monoclinic structure belonging to the C2/c space group is indicated by the XRD pattern of CuO nanoparticles, which shows diffraction patterns of (110), (11), (111), (11), ((20), (020), (202), (11), (31), (113), (311) and (004) at 2θ angles of 32.54, 35.56, 38.81, 38.51, 46.33, 48.82, 53.51, 58.38, 61.62, 66.20, 68.11, 72.54, and 75.19. There is strong agreement between the diffraction data and the CuO JCPDS card (JCPDS 00-101-1194). A = 4.6700 Å, b = 3.4300 Å, and c = 5.1200 Å are the lattice parameters determined from the XRD data. The fact that the peak has become broader suggests that the CuO nanoparticles are quite tiny (Sagadevan, 2017). Further evidence that CuO NPs were produced in a single phase was the absence of any pattern associated with impurities (Vinothkumar, 2019).

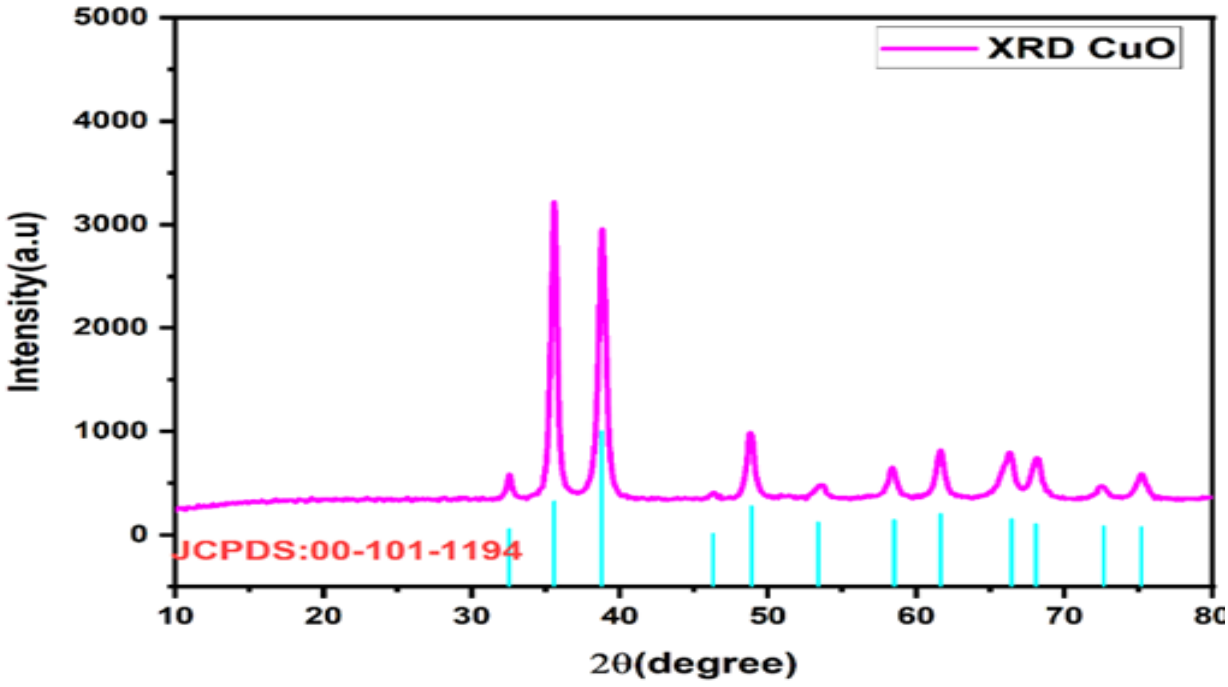

Figure1 *XRD of CuO nanoparticles*

According to Radhakrishnan (2014), Scherer's equation was used to estimate the average crystalline size of the copper oxide nanoparticles.

$$D = \frac{K\lambda}{\beta cos\theta} \quad (1)$$

The formula is as follows: D is the average crystallite dimension, β is the broadening at half bandwidth, λ is the wavelength, and K is a constant that is connected to the shape of the crystallites and the definitions of both β and D; it is approximately equal to one. The results show that 12.85 nm is the average size of the crystallites. Table 1 also displays the results of XRD examination for chemically synthesized CuO NPs, including 2θ angles (positions), miller indices, d spacing, full width at half maximum (FWHMs), crystallite sizes, and average crystallite size. Sagadevan (2017) and others have observed similar findings.

Table 1

2θ angles, d spacing, miller indices, FWHM, crystallite sizes, and average crystallite size of CuO NPs obtained from XRD analysis.

2Theta	Miller indices(hkl)	d spacing(Å)	FWHM	Crystal size(nm)	Average size(nm)
32.54592	(110)	2.748389075	0.43491	19.0211556	12.845
35.55879	(11$\bar{1}$)	2.521589634	0.52851	15.77931107	
38.81174	(111)	2.317577721	0.63993	13.15686795	
46.33125	(11$\bar{2}$)	1.957393053	0.49866	17.32160999	
48.82216	(20$\bar{2}$)	1.863218615	0.65094	13.39712934	
53.51744	(020)	1.710144733	0.91783	9.689929327	
58.38238	(202)	1.578815825	0.78606	11.57210417	
61.62318	(11$\bar{3}$)	1.503340225	0.75313	12.27688595	
66.20172	(31$\bar{1}$)	1.409993106	1.16139	8.162243781	
68.10999	(113)	1.375028324	0.89919	10.65961869	
72.53527	(311)	1.301574653	0.85088	11.57588498	
75.19386	(004)	1.262137788	0.86932	11.52903688	

Morphological study

As shown in Figure 2, the SEM picture of the CuO nanoparticles is presented. Along with a very homogeneous, well-defined crystalline structure, the particles were almost evenly distributed over the image's sphere of view. It was also common for agglomerations to happen more often. Round nanoparticles with a narrow size distribution make up the product. Identical scanning electron micrographs of CuO-NPs have been documented in previous works (Vinothkumar, 2019; Sagadevan, 2017).

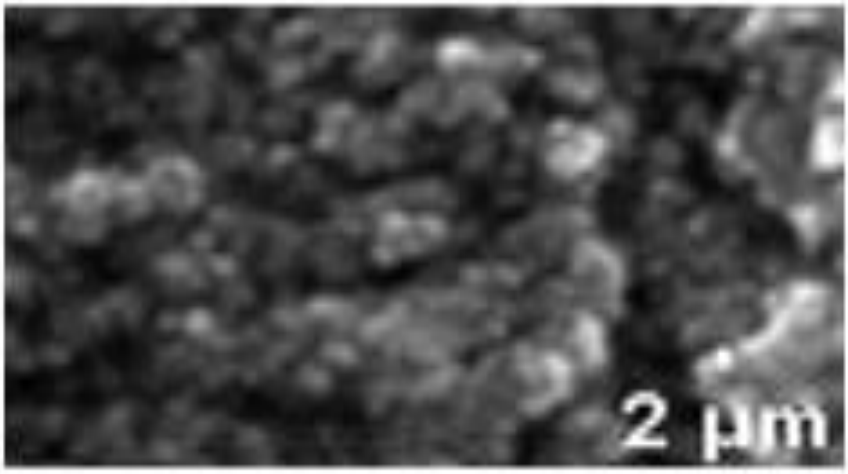

Figure 2 *SEM micrograph of CuO nanoparticles*

Optical studies

Using a UV-Vis spectrophotometer, the optical analysis was conducted. Wavelength versus absorption of CuO nanoparticles is illustrated in Figure 3. The absorption band of CuO NPs, as previously reported (Dhineshbabu, 2016; Sagadevan, 2019), correlates to the wavelength at which CuO nanoparticles exhibited good absorption.

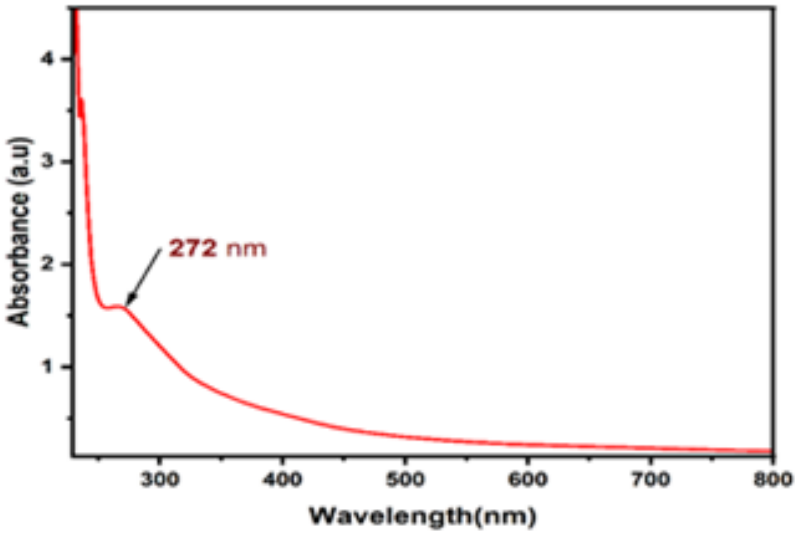

Figure 3 *UV-Vis absorption of CuO nanoparticles*

The optical band gap energy (Eg) of the obtained CuO nanoparticles is calculated from Tauc's relationship (Reena, 2020):

$$(h\upsilon) = A(h\upsilon - E_g)^{1/n} \quad (2)$$

where α is the absorption coefficient, A is constant, υ is the frequency of the photon, hυ is the energy of the incident photon, and n is the exponent that determines the type of electronic transition causing the absorption and can take the values 1/2 and 2 depending on whether the transition is direct or indirect, respectively.

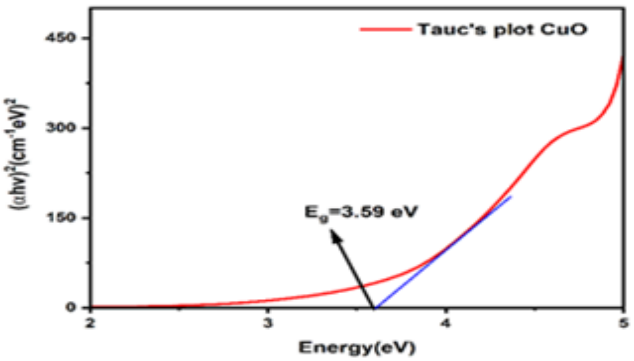

Figure 4 *Band Gap of CuO nanoparticles*

Nanoparticles of copper oxide have a much wider band gap than bulk copper oxide. The presence of an energy intercept of 3.59 eV, as shown by the extension of the linear component of the curve to (αhυ) 2 = 0, indicates this. Quantum confinement is responsible for the sample's larger band gap. According to previous research (Lanje, 2010; Dhineshbabu, 2016; and Sagadevan, 2017), this bandgap energy is within the acceptable range. Quantum confinement, brought about by the nanoparticles' small size or reduction in dimensional structure, leads to dramatic blue shifts because of their high surface area to volume ratio.

CONCLUSIONS

The co-precipitation approach is a simple and inexpensive way to synthesize copper oxide nanoparticles (CuO-NPs). This process necessitated complex machinery and affordable precursors. The XRD pattern revealed that the CuO-NPs had a crystalline and monoclinic shape. Annealed CuO-NPs at 600˚C display the sharpest diffraction peaks and the maximum intensity, with a crystallite size of 12.850 nm. The scanning electron micrograph of the CuO-NPs revealed perfectly round particles. We used ultraviolet (UV) absorption to study the optical characteristics of CuO nanoparticles. The CuO nanoparticles' straight band gap was determined to be 3.59 eV.

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DECLARATION

The authors declare that they have no conflicts of interest.

DATA AVAILABILITY STATEMENT

All data generated from the field experiments and reported in the manuscript are included in the article. Further data sets are available from the corresponding author upon request.

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