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Preparation and properties of Copper Oxide Nanoparticles through the aqueous precipitation method

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Abstract

The Copper nanoparticles are mainly utilized as an antimicrobial agent. They are used in hospitals due to their antimicrobial ability to kill more than 99.9% of Gram-positive and negative bacteria within 2 hours of exposure, if a suitable dose is applied. In this investigation, CuO NPs were prepared through the co-precipitation method. CuO NPs were characterized by X-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM) and Photoluminescence studies. The different bond frequencies associated with the samples were assigned from their Fourier Transform Infra-Red (FTIR) spectra. The optical studies were explained by their UV-VIS and Photoluminescence spectra.

Keywords: CuO NPs, antimicrobial agent, XRD, FESEM, FTIR, UV-VIS, Photoluminescence spectra.

1. Introduction

The field of nanomaterials includes subfields that develop or study materials with unique properties that result from nanoscale dimensions. Surface science and colloidal science have created many materials useful for nanotechnology[1], such as carbon nanotubes and other fullerenes, various nanoparticles and nanorods. Nanoscale materials

can also be used in large numbers of applications. Most of the current commercial applications of nanotechnology are in this flavor [2]. Advances have been made in using these materials for medical applications. See Nanomedicine. Nanoscale materials are sometimes used in solar cells[3, 7], which is competing with the cost of traditional silicon solar cells. Development of applications containing semiconductor Display

technology, lighting, solar cells, biological imaging like nanoparticles used in next-generation products[5]. Copper oxide particles have been found to have mainly utilized the antimicrobial agent as well as superior UV blocking properties compared to their bulk alternatives[6, 8]. These nanoparticles are hard and give them their properties.

2. Experimental and Characterization Methods

2.1 Synthesis

Copper oxide nanoparticles can be synthesized using the aqueous precipitation method. In this method, copper acetate is used as a precursor and sodium hydroxide as a stabilizing agent. Single phase monoclinic structure of the copper oxide nanoparticles is revealed using X-ray diffraction.

The rectangular morphology of the copper oxide nanoparticles is revealed using the scanning electron microscopy. CuO nanostructures were synthesized by precipitation method using copper chloride (CuCl_2) and copper nitrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$). First, each precursor was dissolved in 100 ml deionized water to form 0.1 M concentration. NaOH solution (0.1 M) was slowly dropped under vigorous stirring until pH reached to 14. Black precipitates were obtained and repeatedly washed by deionized water and absolute ethanol for several times till pH reached 7. Subsequently, the washed precipitates were dried at 80 °C for 16 h. Finally, the precursors were calcined at 500 °C for 4 h. investigated by X-Ray Diffractometry (XRD). The morphology was monitored by scanning electron microscope (SEM). Chemical properties were investigated by Fourier transform infrared spectroscopy.

Chemical Data	
Chemical symbol	CuO
CAS No.	1317-38-0
Group	Copper 11 Oxygen 16
Electronic configuration	Copper $[\text{Ar}] 3d^{10} 4s^1$ Oxygen $[\text{He}] 2s^2 2p^4$

Chemical Composition	
Element	Content (%)
Copper	79.87
Oxygen	20.10

2.2 Physical Properties

The physical properties of copper oxide nanoparticles are given in the following table.

Properties	Metric	Imperial
Density	6.31 g/cm ³	0.227 lb/in ³
Molar mass	79.55 g/mol	

2.3 Characterization Techniques

The CuO NPs were characterized by X-ray diffractometer (model: X'PERT PRO PANalytical). The diffraction patterns were recorded in the range of 20°-80° for the CuO NPs sample where the monochromatic wavelength of

1.54 Å was used. The sample was analyzed by Field Emission Scanning Electron Microscopy (Carl Zeiss Ultra 55 FESEM) with EDAX (model: Inca). The FT-IR spectra were recorded in the range of 400-4000 cm⁻¹ by using Perkin-Elmer spectrometer. The absorption spectrum of CuO NPs was studied in the range between 190 and

1100 nm by Lambda 36spectro photometer. Photoluminescence spectra were taken using spectrometer JASCO Spectrofluorometer FP-8200.

3. Results and Discussion

3.1 X-ray diffraction Studies

The X-ray diffraction patterns of the prepared samples are shown in Fig. 1 The XRD peaks are located at angles (2θ) of 31.849, 34.508 and 36.334 corresponding to (100), (002) and (101) planes of the CuO NPs. Similarly, other peaks found at angles (2θ) of 47.614, 56.668, 62.932, 66.45, 68.023, 69.106, 72.61 and 77 are corresponding to (102), (110), (103), (200), (112), (201), (004) and (202) planes of CuO NPs. The standard diffraction peaks show the hexagonal wurtzite structure of CuO NPs with space group $P6_3mc$. It is also confirmed by the JCPDS data (Card No: 36-1451).

This is the most stable phase of CuO NPs. The lattice constants 'a' and 'c' of the *wurtzite structure* of CuO can be calculated by using the relation [1],

$$\frac{1}{d^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}$$

with the first order approximation ($n = 1$) for the (100) plane. The lattice constant 'a' is obtained through the relation $a = \frac{\lambda}{\sqrt{3} \sin\theta}$ and lattice constant

'c' is derived for the plane (002) by the relation $c = \frac{\lambda}{2 \sin\theta}$. The calculated values of 'a' and 'c' are

3.2418 Å & 5.1941 Å for CuO NPs.

The average crystalline size of the samples is calculated by Debye Scherrer's relation

$$\text{Average crystalline size } D = \frac{k\lambda}{\beta \cos\theta}$$

Where, λ - the wavelength of the radiation (1.54056 Å for CuK radiation), k - a constant which is equal to 0.94, β - the peak width at half-maximum intensity, θ - the peak position. The average crystalline size is 33nm for CuO NPs.

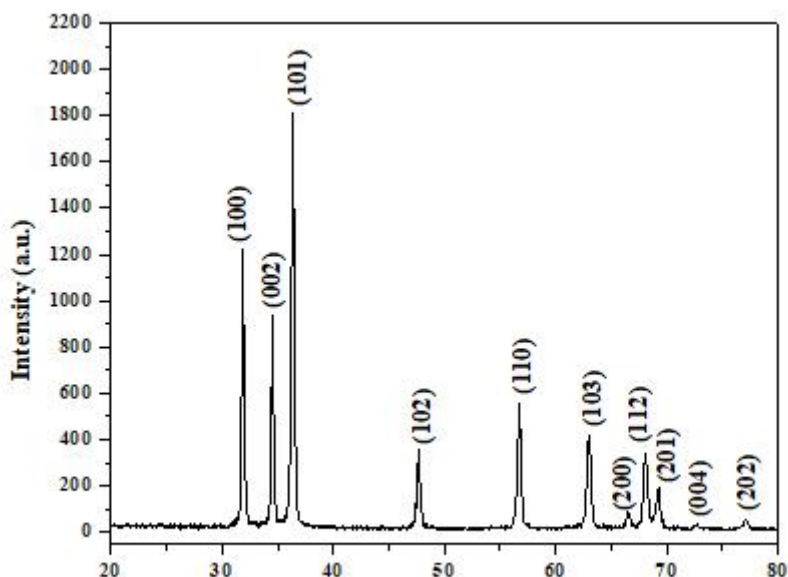


Fig.-1 X-Ray Diffraction Pattern of CuO NPs

3.2 Field Emission Scanning Electron Microscopic Studies

The FESEM is one of the promising techniques for the topography study of the sample and it gives important information regarding the growth mechanism, shape and size of the nanoparticles.

The surface morphology of the CuO NPs is shown in Fig. 2. The entire FESEM image clearly shows the average size of the NPs in the order of nanometer size. The CuO NPs are tree like structure and top of tree uniform even grain boundaries formed. The uniform grain boundaries size 98 nm are found for the CuO NPs.

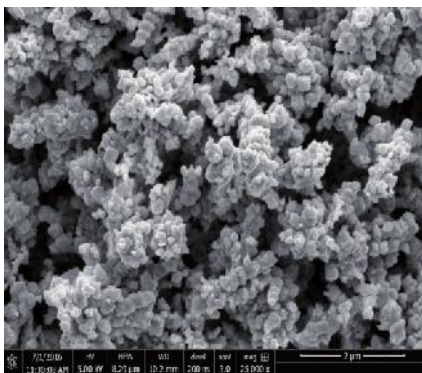


Fig.-2FESEM Image of CuO NPs

3.3 Fourier Transform Infra-Red (FT-IR) spectroscopic analysis

FTIR analysis of CuO nanoparticles The FTIR spectrum (Figure.3) shows bands at around 601,508 and 487 cm⁻¹, which can be assigned to the vibrations of Cu(II)-O bonds. There is sharp

peak observed at 601 cm⁻¹ in the spectrum CuO nanoparticles which is the characteristics of Cu-O bond formation. The broad absorption peak at around 3430cm⁻¹ is caused by the adsorbed water molecules since the nano crystalline materials exhibit a high surface to volume ratio and thus absorb moisture.

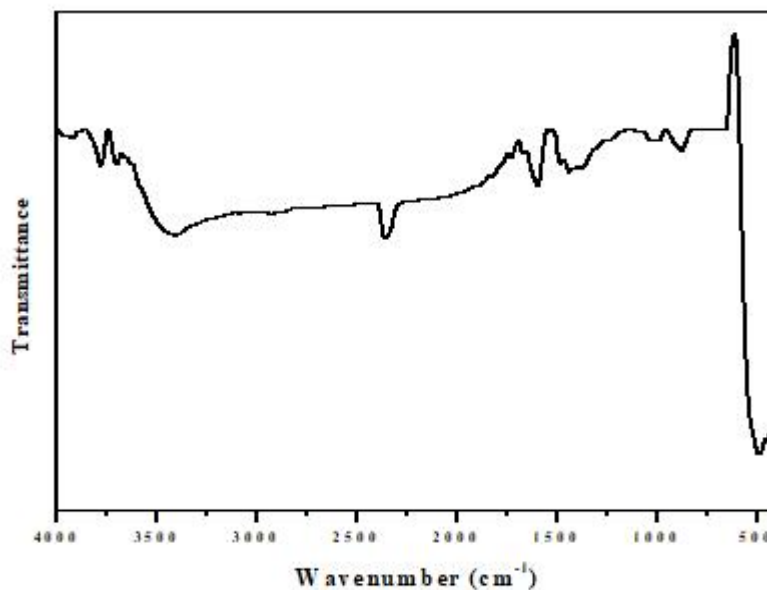


Fig. 3 FTIR spectra of the CuO NPs

3.4 UV-visible absorption spectroscopy studies

Figure-4 shows the UV-visible absorption spectrum CuONPs. The CuO samples absorption spectrum sharp peaks at 372 nm were observed, which believe to arise from the near band edge free excitons. The CuO NPs are expected to show a small red-shift in comparison to bulk CuO. The

band gap energies E_g , of CuO NPs were found 3.3 eV. Showed the small 'red shift' of 0.07 eV from standard bulk band gap at room temperature ($E_g = 3.37$ eV). Where ZnO NPs result in decrease to deviating from the Burstein-Moss shift [3, 4] the estimated band gap values are close to the bulk CuO, this gives conclusion that there is no indication of quantum size effect [5].

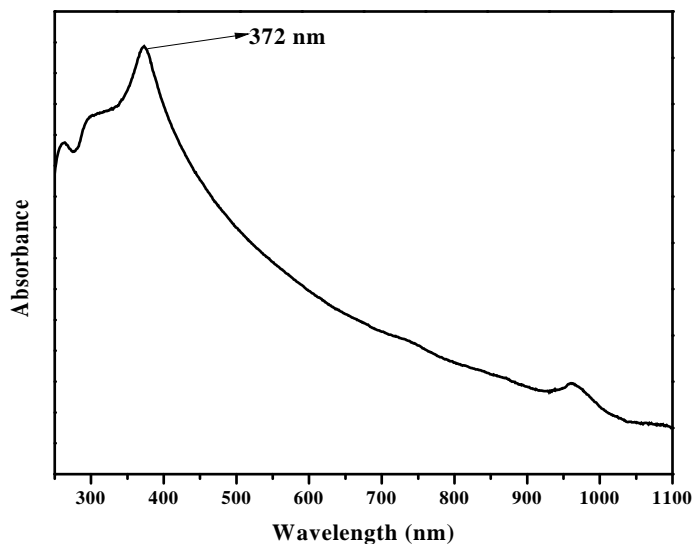


Fig.-4 UV-visible spectra of CuO NPs

3.5 Photoluminescence studies

The photoluminescence spectrum of the CuONPs is shown in Fig.4. The photoluminescence spectra of CuO NPs samples recorded with the excited wavelength of 325 nm. The emission spectra of the CuO NPs sample having six peaks at 395nm, 426nm, 440nm, 469nm, 483nm and 494nm. These bands are Near Band Edge emission, Violet emission, three blue emission and blue-green emission respectively. The NBE emissions is located at UV region 395nm for pure CuO NPs, this NBE emission is attributed to the radiative

recombination between the electrons in the conduction band and the holes in the valence band. The green emission centered at 426 nm, and is ascribed to an electron transition from a shallow donor level of the natural copper interstitials to the top level of the valence band [5]. The three blue emission bands at 440 nm, 469 nm and 483 nm are attributed to singly ionized copper vacancies [6]. There is blue-green emission band at 494 nm due to a surface defects in the CuO NPs corresponding to the transition between oxygen vacancy and oxygen interstitial defect.

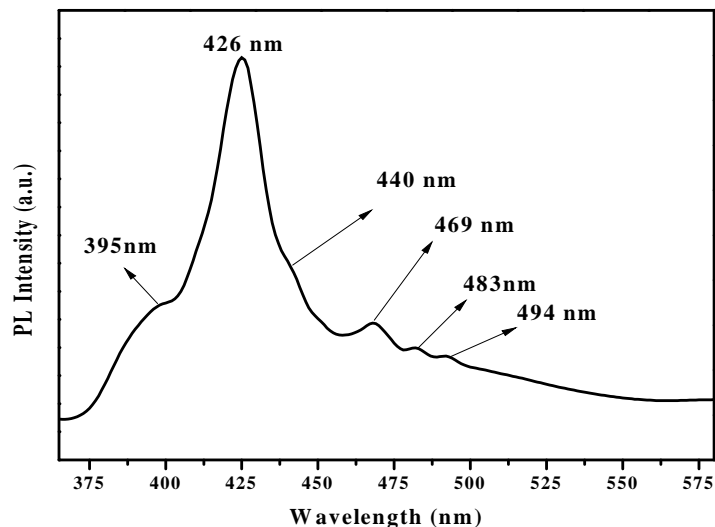


Fig. 5 -Photoluminescence emission spectra CuO NPs

4. Conclusions

The CuONPs were prepared through the co-precipitation method. The X-ray diffraction study confirmed that the prepared particles were in a hexagonal wurtzite structure. The values of lattice parameters 'a' and 'c' were found to be 3.2418 Å & 5.1941 Å respectively for CuO NPs. From the FE-SEM images, the CuO was a tree-like structure and at the top of the tree, even uniform grain boundaries formed. The uniform grain boundaries of 98 nm were found for the CuO NPs. From the FE-SEM analysis, the chemical compositions were estimated. Using the recorded FT-IR spectra, the various vibrational frequencies were assigned to the CuO NPs. From UV-Vis spectra, the CuO sample absorption spectrum sharply peaks at 372 nm. The photoluminescence studies showed that the CuO sample the band emission, which is due to copper vacancies, oxygen vacancies and surface defects.

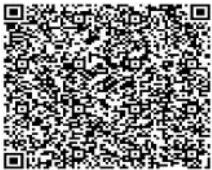
Conflicts of interest

The authors declare no conflict of interest.

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