



## Synthesis of CuO NPs by Using Chemical Method & Biological Evaluation

ELLA. RAJESH<sup>1</sup>, Prof .B. VENKATESWARA RAO<sup>2</sup>, Dr. M.PADMA<sup>3</sup>, Dr.CH.MADHU<sup>4</sup>

Department of engineering chemistry, Andra University, Visakhapatnam - 530003

### Abstract:

In this study, Copper oxide nps were promptly synthesized from  $\text{Cu}(\text{NO}_3)_2$  solution and formation of nps observed with in chemical method. Characterize Copper oxide nps using IR, Scanning electron Microscope (Fe-SEM), AND X- ray diffract meter. Additional its antimicrobial activity against bacillus subtilis and, Escherichia coli is studied.

**Key words:** Nanoparticles, Copper oxide, bacillus subtilis, Escherichia coli.

### 1 INTRODUCTION

Nanotechnology obtained a great extent of attention for its imperative pioneering role in exploiting materials at the atomic and molecular levels to radically adjust the product properties. Materials consized to the nanometric scale display remarkably different properties compared to what they display at the macroscale or microscales. Because of their unique properties, nanomaterials are widely used in a diversity of applications. Small amounts of nanoparticles can play an essential role in developing the properties of materials. Nanoparticles are becoming more and more important day by day as they play a favourable role in a wide variety of scientific fields. In general, the size of nanoparticles spans the range between 1 and 100 nm. Nanotechnology comprises the design, construction, and utilization of functional structures with at least one characteristic dimension measured in nanometres. Currently, nanoparticles are widely using in many fields [1-2]. Transition metals are large in number and have diverse applications in numerous fields. CuO is one of the useful metal oxide and which has many applications in different fields. Copper oxide nanoparticles are of special interest because of their efficiency as nanofluids in heat transfer applications. It has been reported that a 4% addition of CuO improves the thermal conductivity of water by 20% [3]. CuO-NPs have been prepared with different sizes and shapes via several methods such as sonochemical [4], direct thermal decomposition [5], electrochemical methods [6], colloidthermal synthesis process [7], and microwave radiation [8]. In the present study, we have synthesized CuO nanoparticles by simple aqueous precipitation method. The copper oxide nanoparticles (CuO-NPs) possess a huge range of applications. Compared with ordinary copper oxide powder, the nanoparticles of copper oxide exhibit superior catalytic activity and selectivity[9]. It has excellent antimicrobial activity against various bacterial strains. The CuO-NPs were used in the removal of dyes. The CuO-NPs also have an application in heat transfer[10]. The thermal conductivity of CuO based nanofluid is 12.4% higher in comparison with deionized water[11]. This method involves a simple, cheap, and one-step process for the synthesis of CuO nanoparticles.

### 2. MATERIALS AND METHOD

#### 2.1 MATERIALS

All of the chemicals used in the experiment are of analytical grade and used as purchased without any purification. Copper nitrate trihydrate ( $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ ), of 98% purity is used. De-ionized water is used as solvents. Sodium hydroxide (NaOH) is used to adjust the pH.

#### 2.2. SYNTHESIS OF COPPER OXIDE NANOPARTICLES

To the first Copper nitrate trihydrate  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  and sodium hydroxide NaOH were each dissolved separately in deionized water to form the liquid media of the desired concentrations of 0.05M (12.85g/500mL) and 0.1M (2g/500mL) for sample A and B respectively the ratio of the concentrations was 1:1 ( $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ : NaOH). The copper nitrate trihydrate was slowly added dropwise to NaOH solutions under vigorous stirring at room temperature, forming transparent solutions, then inserted into an electrical oven at 90°C for 2 hours. These solutions were reacted to produce copper oxide precipitates. Following the precipitation, the solution was centrifuged at 3000 rpm for 30 minutes. The supernatant was then removed, and the precipitate which contains copper oxide was obtained. Finally, the copper oxide was ground with mortar to be shaped into powder.

### 2.3. CHARACTERISATION OF NANOPARTICLES

#### 2.3.1. UV Absorption and Reflection Spectra

It was carried out by measuring the diffuse reflectance spectroscopy in the UV-Vis range. The spectrum was taken in the range of 200-800 nm. Fig1 shows the diffused reflectance spectra (absorbance as a function of wavelength). The exciton absorption is at about 260 nm. The optical band gap  $E_g$  of the nanoparticles was calculated from the Tauc plot as shown in Fig.2 the presence of a single slope in the plot suggests that the films have direct an allowed transition. For such transition we have

$$(\alpha h\nu)^2 = A(h\nu - E_g)^n \dots\dots\dots (1)$$

Where  $\alpha$  is absorption coefficient,  $h\nu$  is photon energy,  $E_g$  is optical band gap,  $n$  is 1 for direct transition &  $A$  is a constant. Fig.2 shows the bandgap energy is obtained by extrapolating the straight-line portion of the plot to zero absorption coefficient. The bandgap value of CuO nanoparticles is found to be 2.26 eV. This redshift of the bandgap energy is due to the agglomeration of the nanoparticles into larger particles as reported by various authors in different kinds of literature [28-31].

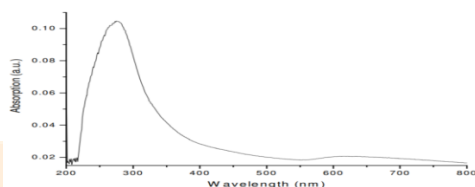


Fig.1: UV-VIS absorption spectrum of as-synthesized copper oxide NPs

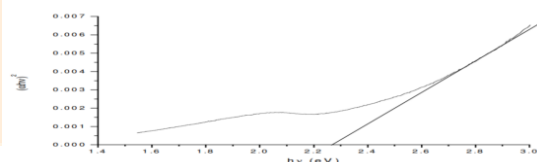


Fig.2. Tauc plot for as-synthesized copper oxide NPs

#### 2.3.2. FTIR Spectrum of Copper Oxide Nanoparticles

FTIR spectroscopy uses a Michelson interferometer to produce an interferogram and the spectrum of CuO is shown in Fig.3. It has been shown that as particle size decreases, an increase in frequency for the bond is observed in nanoparticles. Bands at  $416.35\text{ cm}^{-1}$  are assigned to the stretching vibrations of Cu-O. The bulk CuO stretching frequency is observed at  $424\text{ cm}^{-1}$ . Here a blue shift is observed in that frequency i.e., that frequency is due to quantum confinement. Three intense bands were plotted at  $1384.34\text{ cm}^{-1}$ ,  $1041.54\text{ cm}^{-1}$ , and  $1556.58\text{ cm}^{-1}$  and are attributed to the stretching vibrations of C = O, C = C, and C-H groups in acetate species, which suggests its presents as adsorbed species in the surface of nanoparticles. The broad absorption peak centered at  $3423.61\text{ cm}^{-1}$  and  $1626.40\text{ cm}^{-1}$  correspond to O-H stretching and bending frequencies of  $\text{H}_2\text{O}$ , indicating the existence of water on the surface of nanoparticles [32]. Variations of the peak positions of CuO [33-35] are presented in Table .1.

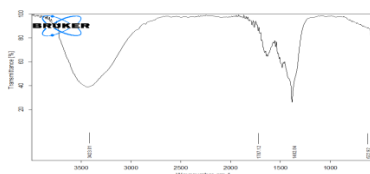


Fig 3 FTIR Spectra of Copper Oxide nanoparticles

The peak observed at 622.92 cm<sup>-1</sup> is the presence of copper oxide nanoparticles. The broad absorption peak centered at 3423.01 cm<sup>-1</sup> corresponds to O-H stretching of water indicating the existence of water on the surface of nanoparticles.

S.No	CuO (cm <sup>-1</sup> )	Vibrational modes
1	3423.01	OH
2	1482.84	OH deformation
3	622.92	Stretching of CuO
4	1781.01	C-H

Table.1. Comparison of Vibrational modes of copper oxide nanoparticles

### 2.3.3 Powder XRD analysis

Fig.4 shows the crystal structure and purity of the synthesized CuO nanoparticles were determined by powder XRD. The prominent diffraction peaks at 2θ values 30.89°, 33.92°, 35.65°, 56.32°, 62.53°, 66.72°, 68.87°, 76.07° are associated with [110], [111], [022], [202], [113], [020], [220], [222] planes respectively. The evolved diffraction peaks could be indexed to a monoclinic phase of CuO with lattice parameters a= 4.683 Å, b= 3.421 Å, c= 5.129 Å and corresponds to the JCPDS file no 80-1268. The peak intensities and width of the spectrum indicates the presence of nanoscale crystallites. The absence of peaks corresponding to by-products such as Cu(OH)<sub>2</sub> or Cu<sub>2</sub>O indicates the phase purity of CuO nanoparticles.

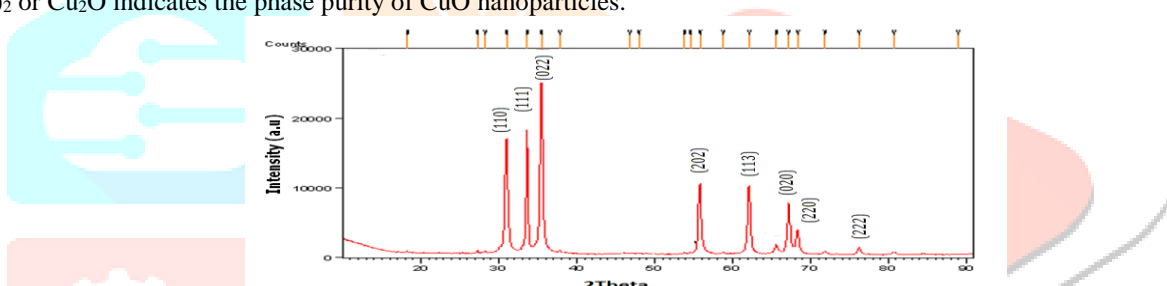


Fig.4. XRD spectra of Copper oxide nanoparticles

2θ	θ	Sinθ	d(Å)	Nm	HK
30.89	15.44	0.266	2.893	77.01	110
33.92	16.96	0.291	2.641	85.02	111
35.65	17.82	0.305	2.517	86.24	022
56.32	28.16	0.471	1.632	73.55	202
62.53	31.26	0.518	1.484	74.93	113
66.72	33.36	0.549	1.401	70.09	020
68.87	34.43	0.565	1.362	65.06	220
76.07	38.03	0.615	1.250	70.92	222

Table2. d-spacing calculations for copper oxide NPs

Inter planar d-spacing was calculated using Bragg’s Law equation (Table. 2):

$$2d\sin\theta = n\lambda \dots\dots\dots (2)$$

where θ is Bragg’s angle of diffraction, λ is X-ray wavelength, i.e. 1.5406 Å, and n = 1. Further, particle size was calculated from the intense peak corresponding to (101) plane using Debye–Scherrer formula. The crystallite size is calculated using the Debye Scherrer equation

$$d = 0.9 \lambda / \beta \cos\theta \dots\dots\dots (3)$$

Where  $d$  is the average crystallite size (nm),  $K$  is the grain shape factor (0.9),  $\lambda$  is the X-ray wavelength (nm),  $\beta$  is the line broadening at half the maximum intensity in radians, and  $\Theta$  is the Bragg diffraction angle of the  $2\Theta$  peak. The average crystallite size was estimated to be 20-50nm.

#### 2.3.4. Field emission Scanning Electron Microscope (FESEM)

FE-SEM image Fig.5. shows the surface morphology and particle size of the synthesized copper oxide nanoparticles. It is clear from the images that the size of the copper oxide nanoparticles is ranging from 20-5nm. The obtained products are composed of near flower-shaped morphology with the average size in the range of 50 nm.

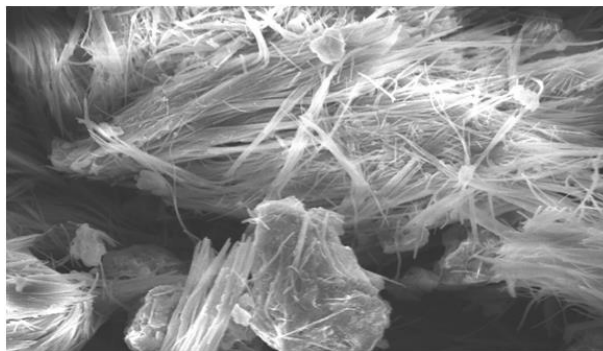


Fig.5. FESEM image of copper oxide nanoparticles

#### 2.3.5. Energy Dispersive X-ray Spectroscopy (EDX)

EDX spectrum, Figure.6, plot not only identifies the elements corresponding to each of its peaks but the type of X-ray to which it corresponds as well. The higher a peak in a spectrum, the more concentrated the element is in the spectrum. Spherical shaped morphology is observed in the micrograph of copper oxide nanoparticles.

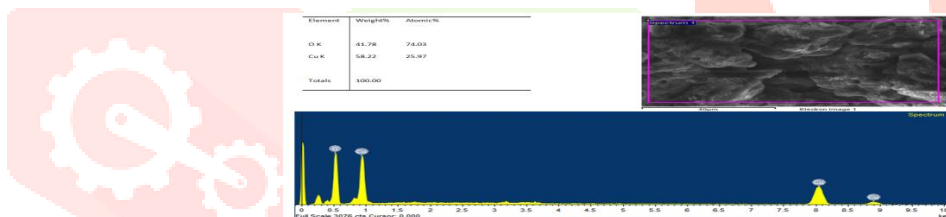


Fig.6.EDX analysis of copper oxide nanoparticles

The dried powder of the sample was analyzed on Energy Dispersive X-ray Analysis (EDX) technique. The peaks have confirmed the presence of copper, oxygen. The average atomic weight percentage ratio of Cu, O. CuO nanoparticle was 58.22: 41.78. The energy ratio (in keV) 25.97: 74.03. The data are presented in Fig.6. The presence of doped rare earth elements in copper oxide nanoparticles was confirmed by the analysis.

#### 2.3.6. Antimicrobial Screening of copper oxide

The nanosized copper oxide particles are screened in vitro for antibacterial activity against *E. coli*, *B. subtilis*, and antifungal activity against *A. niger* by the Agar-well diffusion method. The antibacterial and antifungal activities of copper oxide nanoparticles are listed in the table.3.



Fig.7 Inhibition zones for CuO against. *B. subtilis* *E. coli*



**Fig.8.** Inhibition zones for CuO nanoparticles against *A. niger*

Bacteria	Inhibition zone (mm)
<i>E. coli</i>	08
<i>B. subtilis</i>	08
Fungi	Inhibition zone (mm)
<i>A. Niger</i>	0

**Table.3.** Antimicrobial activities of copper oxide nanoparticles

The copper oxide nanoparticles showed good antibacterial activity against *B. subtilis* and *E. coli* but didn't show antifungal activity against fungal organisms. We found greater inhibition zones for *E.coli* and *B.subtilis* but the inhibition zone for *A.niger* is 0 mm which means it doesn't exhibit any activity.

In the present study on the "synthesis of copper oxide nanoparticles using the chemical method and their antifungal activity", copper oxide nanoparticles were synthesized using copper nitrate trihydrate and NaOH. Synthesis conditions were optimized and resultant nanopowder was characterized using UV- Visible spectroscopy, XRD, FESEM, FTIR, EDX. The morphological analysis reports a particle size range of 20-50 nm and also revealed that the nanoparticles are present in the form of aggregates. In vitro studies were carried by Disc Diffusion method. The bacterial organisms used in this study are *Escherichia coli*, *Bacillus subtilis*. The observed inhibition zones for these nanoparticles are in the range of 10-15 mm for *E.coli* and 10-17 mm for *Bacillus Subtilis*. Where as anti fungal activity is also carried on *A.niger* where it did not show any activity. The screened data in these reports are in good agreement with the previous data and the inhibition zone images are pictorially recorded. This study conclusively reports a synthesis of copper oxide nanoparticles. Such studies have the potential for developing good bactericidal formulations having nanoparticles.

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