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# Structural, Optical, Morphological and Thermal Properties of CuO Nanoparticles Prepared by Sol-gel Technique

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This article aims to report an environmental friendly preparation technique of copper oxide nanoparticles, their structural and morphological properties, and band gap energy analysis. Copper oxide (CuO) nanosize particles are prepared by sol-gel route. Powder X-ray diffraction, Fourier transform infrared spectroscopy, scanning electron microscopy, UV-visible absorbance spectra and thermal spectra are used to analyze the nanoparticles. Powder XRD analysis showed that monoclinic structure with excellent crystallite size and *d*-spacing distance was found. FTIR spectral results confirmed the presence of the Cu-O bands in prepared nanoparticles. Morphology of the samples indicates and confirms the random spherical shape of nanoparticles. Prepared CuO particles show that a color change occurs in synthesis and confirm its respective peaks at 253 nm, which were analyzed through UV-Vis spectroscopy. The band gap is determined equal to 1.3 eV. The exothermic and endothermic processes of prepared nanoparticles were investigated by TG/DTA experiments.

**Keywords:** Nanoparticles, Precipitation, Wavelength, Thermal, Optical.

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## 1. INTRODUCTION

Nanocrystalline materials are of great interest and attract attention due to their properties that differ from those of molecules and bulk materials [1]. Because their application depends on nanomaterial shape, size and structure, understanding of nanomaterials as well as a new synthesis method is brilliant in nanotechnology [2]. In recent years, nanosize materials have specific properties such as high surface-to-volume ratio, optical and thermal properties as compared to bulk materials. As an effect of their size in the nanometer scale and the special light emission and absorbance properties, a change of band gap is discussed when the particle size is reduced [3]. In semiconductors, the band structure of the nanoparticles is controlled by quantum effect, and many effects can be changing the size of particles. Recent research in semiconducting nanosize particles is major investigation due to their wide properties [4].

The many number of metal oxides is available in common world, but some special types of the metal oxides are most useful application in everyday life in technology. Many transition metals are in the periodic table and have many fields in different types of applications. Some examples of metal oxides like $MnO_2$, ZnO and SnO have proved as high potential applications in several field [6]. In the normal technological world, CuO is also one of the most useful metal oxides, which has a large number of applications in many fields. CuO nanoparticles are uniqueness behavior even though like bulk or nanosize [6]. The transition type of metal oxides is cupric oxide (CuO) [7]. The CuO is a narrow *p*-type semiconductor. CuO nanoparticles are of very much important scientific interest because of their potential applications in sensing catalysis, cosmetics, and optoelectronic devices [8]. Copper oxide (CuO) is one of the most important metal oxides, which has applications in many fields [9]. The efficiency of CuO nanofluids has heat transfer applications, so CuO is a very special nanoparticle [10]. However, the reports on the synthesis and characterization of CuO nanoparticles are most important application related to some other metal oxides such as ZnO, TiO, SnO and FeO, is a very small band gap semiconductor. The size, physical and chemical properties of CuO nanoparticles depend on their microstructure such as the morphology [11]. In this paper, we have synthesized copper oxide nanoparticles by simple sol-gel method. The finally prepared CuO nanoparticles were analyzed by powder X-ray diffraction, Fourier transform infrared spectroscopy, scanning electron microscopy, UV-visible spectroscopy and TG/DTA analysis.

## 2. METHODS AND MATERIALS

### 2.1 Materials and Preparation of CuO Nanoparticles

The analytic reagent grade chemicals were used in this experiment. Cu $(CH_3COO)_2$ $H_2O$, NAOH and $CH_3COOH$ were purchased from Merck, India. Deionized water alone was used in the experiment.

The 0.025 M solution of copper acetate is prepared, and 1 ml of acetic acid is added and heated to 40 $^0$C with constant stirring. The NaOH was added during the stirring; prepared solution was till reach at 9 pH value. Finally, we got milky blue powder precipitate, and it was washed with deionized water and then dried in room temperature for one week.

### 2.2 Characterizations

The structure identifications were carried out using powder X-ray diffraction. The presence of functional group in the prepared samples was found using FTIR.

 

Morphology analysis of the samples was carried out by SEM. Absorption spectra of the samples were recorded and calculated by the band gap using UV spectrometer. Finally, thermal analysis was carried out by TG/DTA method.

## 3. RESULTS AND DISCUSSION

### 3.1 Structural Characterization

X-ray diffraction analysis method is the most common tool to analyze the crystal structure and confirm the purity of the prepared nanoparticles. Fig. 1 shows the pattern of powder XRD of as prepared CuO nanoparticles. The four major diffraction peaks were observed in XRD pattern from Fig. 1. According to the JCPDS file number 05-0661, four reflection angles at $2\theta = 38^0$, $48^0$, $61^0$, and $66^0$ were found in diffraction peaks. The monoclinic structure of CuO nanoparticles was confirmed from the diffraction peaks declared as a plane of [1 1 1], [2 0 2], [1 1 3], and [3 1 1]. The crystal cell parameters of CuO nanoparticles are $a = 4.684$, $b = 3.425$, $c = 5.129$. The average crystalline boundary size of CuO is measured around 40 nm from FWHM [12] as calculated by Scherrer equation. The Scherrer equation is [13]

$$D = 0.9\lambda/\beta\cos\theta,$$

where $\beta$ is the full width at half maximum (FWHM), $D$ is the mean crystallite boundary size in nm, $\lambda$ is the wavelength of X-ray (1.5406 Å), and $\theta$ is the degree of the diffraction peak. The FWHM was increased to increasing crystalline boundary size of the nanoparticles [14].

The inter-planar spacing between atoms ($d$-spacing) of the CuO is 4.3 Å, is calculated using Bragg's law. Bragg's law is

$$2d\sin\theta = n\lambda,$$

where $\theta$ is the degree of the diffraction peak in degrees, $d$ is the $d$-spacing of the nanoparticles, $\lambda$ is the wavelength of X-ray, and $n$ is the order of diffraction. The average specific surface area of CuO was $2.38 \cdot 10^{13}$, is calculated by

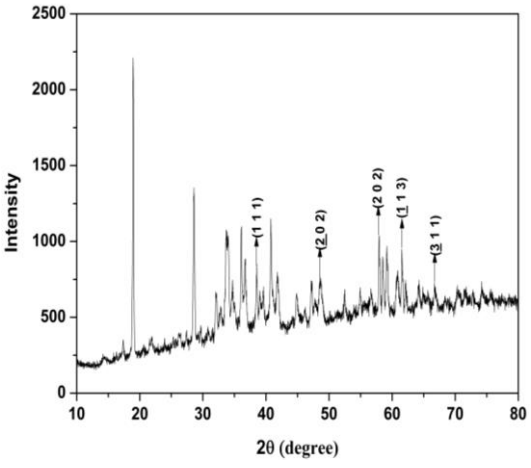


**Fig. 1** – X-ray diffraction pattern of CuO nanoparticles

$$\text{SSA} = 6000/D\rho,$$

where $D$ is the crystalline size in meters, SSA is the mean specific surface area of CuO, $\rho$ is the density of CuO (0.00631) in kg/m$^3$. The crystalline boundary size was increased to increasing specific surface area of CuO.

### 3.2 Fourier Transform Infrared Spectroscopy

The presence of functional groups of the as-prepared CuO powder sample is analyzed by FTIR spectroscopy. Infrared spectra were recorded in the range 400-4000 cm$^{-1}$ on FTIR spectrometer. The obtained spectra are shown in Fig. 2. The FTIR spectra present the peak at around 484 cm$^{-1}$, it was confirmed by vibration bands of Cu(II)-O [13]. There is one sharp peak observed at 654 cm$^{-1}$, it was confirmed by bands of Cu-O. There are other two sharp peaks at 1417 cm$^{-1}$ and 1554 cm$^{-1}$ that may be assigned by O-H bending vibration bond combined with copper atom [15, 16]. A small peak at 2354 cm$^{-1}$ in CuO is due to the presence of atmospheric $CO_2$. The stronger peaks of CuO at 2924 cm$^{-1}$ and 3382 cm$^{-1}$ were observed and confirmed the functional groups. The sample almost shows the same type of functional groups.

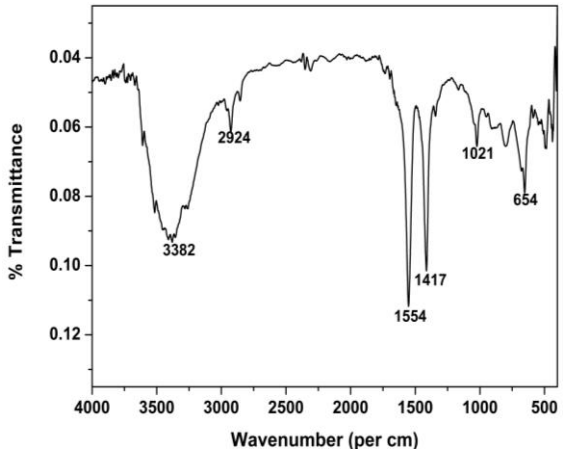


**Fig. 2** – FTIR spectra of CuO nanoparticles

### 3.3 Scanning Electron Microscopy

Scanning electron microscopy (SEM) is a good versatile method available for the analysis of surface morphology and structural studies of nanoparticles. The morphologies of as prepared samples of CuO nanoparticles were investigated through SEM images shown in Fig. 3. SEM measurements were carried out on CuO nanoparticles to uncover morphological differences in the systems at the micrometer scales. It shows the distribution of spherical nanoparticles of the prepared sample of CuO nanoparticles [17]. The SEM images show single rose structure and like confirm randomly spherical shape. The diameter of the CuO nanoparticles at the beginning of the experiment is 38 to 42 nm. The fine nanoparticles formed aggregations due to maximum surface energy. SEM image analysis was confirmed with the help of XRD results.

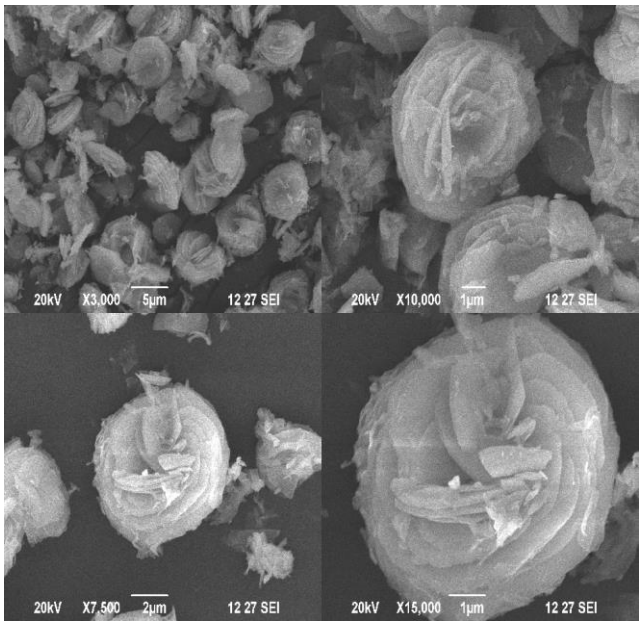


**Fig. 3** – SEM images of the CuO nanoparticles

### 3.4 UV-Visible Spectroscopy

The most useful method is UV-Vis absorption spectroscopy, which is important as it gives fundamental information about the optical properties of the nanomaterial. The room temperature ultraviolet-visible spectrum of CuO nanoparticles samples was recorded. The UV absorption spectrum was used to find electron transition and energy band gap [15]. Fig. 4 shows the absorption spectra of CuO nanoparticles. There are two fundamental strong absorption peaks at 227 nm and 253 nm due to the direct electron transition.

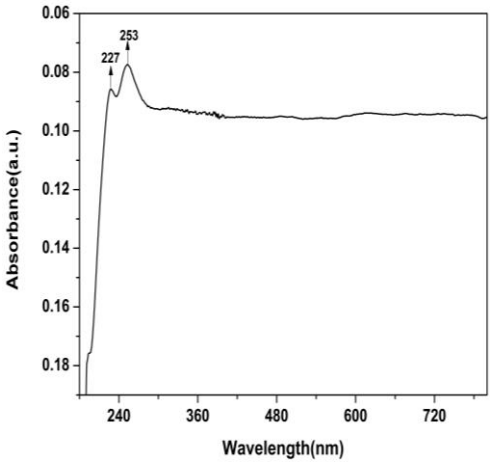


**Fig. 4** – UV-visible spectra of CuO nanoparticles

From the fundamental absorption, the electron transition from valence band to conduction band can be used to determine the value of energy band gap. The functional relation between photon energy and $(\alpha h\gamma)^2$ of nanoparticles is shown in Fig. 5. The energy band gap is measured by extrapolating the straight linear part on the $h\gamma$ in $x$-axis. From the XRD results, CuO nanoparticle is a perfect crystalline structure, and confirmed crystalline structure from it. Energy band gap of indirect band gap semiconductor is smaller than that of the direct band gap semiconductor. The calculated band gap is 1.3 eV, which was smaller than the bulk material band gap (3.5 eV).

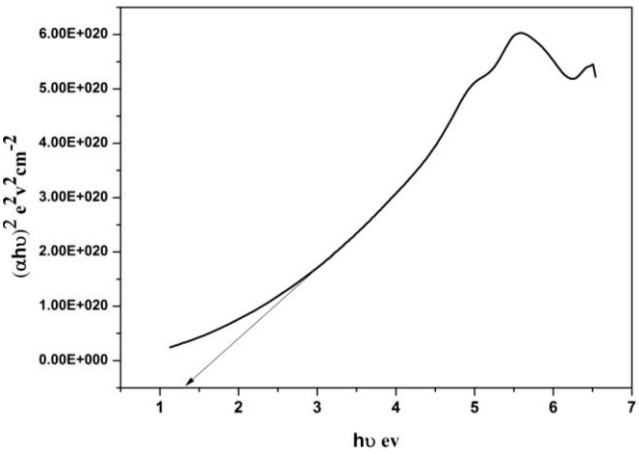


**Fig. 5** – Band gap energy of CuO nanoparticles

### 3.5 TG/DTA Analysis

Thermogravimetric analysis (TGA) is the most important tool in physical and chemical sciences, and can give much of data about physical and chemical phenomena of prepared nanomaterials. During thermal analysis, as prepared samples can undergo various phase transitions. TG/DTA curves determine and give the details about the temperature, at which the prepared CuO nanoparticles have weight loss. Fig. 6 shows thermal gravimetric analysis and differential thermal analysis (TG/DTA) data of the nanoparticles. TG/DTA analysis of the nanomaterial is performed in the temperatures range 30-800 °C in the normal air atmosphere at the heating rate of 1 $^0$C/min. This weight loss is due to vaporization of water content in the nanoparticles. The first weight loss is found at the temperature of 131 $^0$C; the second, third and fourth weight losses were estimated at the temperatures of 193 °C, 254 °C, and 309 $^0$C. The TG/DTA results found that CuO nanoparticles are highly stable, and they have very less weight loss when compared to bulk samples of CuO.

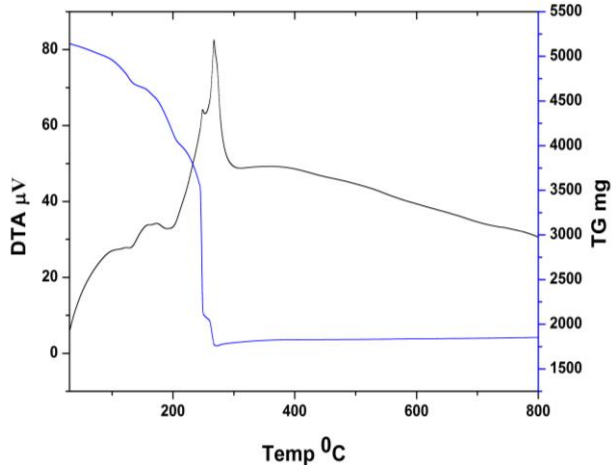


**Fig. 6** – TG/DTA of CuO nanoparticles

## 4. CONCLUSIONS

The synthesis of CuO nanoparticles was carried out via sol-gel method. The crystalline boundary size and structure of prepared CuO nanoparticles were found by powder XRD method. The phase purity and functional groups of the samples were characterized by FTIR study. The morphological studies revealed that the particles were spherical in structure and slightly agglomerated using SEM analysis. The absorption wavelength was 227 and 253 nm from UV spectra, and band energy was also calculated. The weight loss of the nanoparticles was calculated by TG/DTA analysis.

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## Структурні, оптичні, морфологічні та теплові властивості наночастинок CuO, підготовлених методом золь-гелю


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У статті описана екологічна технологія підготовки наночастинок оксиду міді, їх структурні та морфологічні параметри та аналіз енергії забороненої зони. Нанорозмірні частинки оксиду міді (CuO) були виготовлені методом золь-гелю. Для аналізу наночастинок застосовуються порошкова рентгенівська дифракція (XRD), інфрачервона спектроскопія з використанням перетворення Фур'є (FTIR), скануюча електронна мікроскопія, спектри поглинання в ультрафіолетовій і видимій (UV-vis) областях та теплові спектри. Аналіз порошкової XRD показав, що було виявлено моноклінну структуру з відмінним розміром кристалітів та *d*-відстанню. Спектральні результати FTIR підтвердили наявність енергетичних смуг Cu-O у підготовлених наночастинках. Морфологія зразків підтверджує випадкову сферичну форму наночастинок. Підготовлені частинки CuO показують, що зміна кольору відбувається при синтезі, що підтверджують його відповідні піки при 253 нм, які були проаналізовані за допомогою UV-vis спектроскопії. Ширина забороненої зони дорівнює 1.3 еВ. Екзотермічні та ендотермічні процеси підготовлених наночастинок були досліджені експериментами TG/DTA.



**Ключові слова:** Наночастинки, Осадження, Довжина хвилі, Тепловий, Оптичний.