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Facile Synthesis of Spherical Flake-shaped CuO Nanostructure and Its Characterization towards Solar Cell Application

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Abstract

The aim of this work is to synthesis CuO nanoparticles and investigates their eminent properties to identify their diverse application capability in the field of solar cells. In this work, CuO nanoparticles were synthesized by precipitation method using Copper (II) nitrate and Copper (II) chloride. Surface effects due to defects, structural properties related through lattice parameter, and crystallite sizes of nanoparticles have been identified from XRD. The crystal plane and reflection peak position was calculated using Bragg's law. It showed that CuO nanoparticles have a monoclinic structure, and that the average crystallite size of CuO nanoparticles was 28.82 nm. n-type semiconductor behavior had a direct band of E_g = 1.465(2) eV, analyzed from optical studies by DRS. The band gap of the sample was determined from the reflectance spectra using Kubelka-Munk (K-M) function. Elements present were found through absorption peak of FTIR. The blue shifts observed in FTIR spectra in CuO nanoparticles were compared with that of bulk CuO, and absorption band agreed with XRD results. Morphological studies revealed the formation of spherical flake-shaped formation of CuO. It had a higher surface area and was well-suited to solar cell applications.

Keywords: CuO nanoparticles, Precipitation method, Monoclinic, Band gap, Solar cell

Introduction

Metal oxides are crystalline solids that contain a metal cation and oxide anion [1]. A few of the metal oxides are largely beneficial in accordance with their applications in day to day science and technology development. Some transition metal oxides have been proved to be potential candidates for many applications, such as microelectronics, photocatalysis, magnetic devices, powder metallurgy, solar energy transformation, semiconductors, varistors, gas sensors, bio sensors, humidity sensors, mechanical, electrical, and optical switching devices, and antimicrobial agents [2-5]. Nanoparticles and nanostructured materials have received significant interest in latter applications because of their distinguished performance.

Solar cell technology has recently been receiving more attention in research fields because of its major challenges today. The cost of manufacturing solar cell components is very high, making it very expensive. Moreover, the percentage of converted solar power is extremely low. In solar cell technology, $CuInSe_2$ (or its alloys, such as $CuInS_2$ or $CuInGaSe_2$), CdTe, and amorphous silicon materials, CuO or Cu_2O, have band gap in the visible region [6-8], and can be used as its base material. Due to the high light absorption properties of CuO, this material is selected for this study [9].

CuO is a prominent high ionic nano particulate metal oxide. Copper (II) oxide, or cupric oxide, is an inorganic compound, with the formula CuO. It appears as a brownish black powder. Copper oxide is a

compound of 2 elements, copper and oxygen, which are d and p block elements in the periodic table, respectively. In a crystal, a copper ion is coordinated by 4 oxygen ions [10]. CuO is a p-type semiconductor with a monoclinic structure, with giant magnetic field resistance, having a narrow band gap of 1.2 - 1.9 eV, and is used in discrete applications, like gas sensing (detecting large amounts of gas), solar energy conversion, field emission, magnetic storage media, organic catalysis, owing to its photoconductive and photochemical properties [11-14], coating, and pharmaceutical products [4,15]. It is used as a pigment in ceramics to produce blue, red, and green, and sometimes gray, pink, or black glazes. Comparing CuO bulk materials with CuO the nanoparticle, the latter shows better catalytic activity and selectivity. It is also used as a burning rate catalyst in rocket propellant [16]. It is a cheap electrode material for exploring marginal pathways to tailor the functionalities of electrode equipment and construct super capacitors with enhanced energy and power densities [17]. Its synthesis in bulk quantity, due to large applications of CuO, has become an interesting field for many researchers, especially material scientists. Many methods are used to synthesis CuO nanoparticles, which include sol-gel method [18-21], precipitation method [22], electro chemical discharge process [23], solvo-thermal process [24], spray pyrolysis [25,26], sono-chemical reaction [27-30], hydrothermal method [5,31,32], etc.

Phiwdang *et al.* [12] synthesized CuO nanoparticles by precipitation method using different precursors. CuO nanostructures with different shapes, sizes, and morphologies were achieved using different precursors via this process. Aparna *et al.* [18] demonstrated that CuO nanoparticles have potential applications for solar energy transfer, sensors and storage devices, and super conductors. Dahrul *et al.* [20] reported prepared CuO films by sol-gel method. The results indicated that CuO film prepared by sol-gel method has the potential to be developed for solar cells. Mansoury *et al.* [33] investigated the Al_2O_3/water nanofluid performance of parallel flow heat exchangers. Here, double pipe heat transfer acheived the heat transfer coefficient with a maximum enhancement of 26 %. Bashirnezhad *et al.* [34] reported a brief review about nanofluids based on thermal conductivity, in which all types of nanoparticles had reduction in particle size with respect to increase in thermal conductivity. Kherbeet *et al.* [35] investigated the experimental effect on nanofluid by heat transfer characteristics. They observed the Nusselt number for microscale backward-facing step (MBFS) and forward-facing step (MFFS) on the heat transfer. Basically, the Nusselt number increased with decrease in the density of nanoparticle material, decrease in the diameter of nanoparticles, and increase in volume fraction.

In this study, to realize the potential application of CuO, precipitation method is appropriated for bulk synthesis of CuO nanoparticle because it is eco-friendly, highly safe, inexpensive, and has good yield. In addition, the composition, morphology, particle size, and band gap are characterized via X-ray diffraction (XRD), Scanning electron microscopy (SEM), Fourier transform infrared spectrum (FTIR), and Diffuse Reflectance Spectra (DRS). The knowledge obtained should enhance a better understanding of CuO nanoparticles and assists in the further development of novel properties, which may lead to practical industrial application in the future. Moreover, our aim is to change the morphology of CuO nanoparticles to obtain good efficiency in solar cell applications.

Materials and methods

Synthesis of CuO nanoparticles

In a typical synthesis procedure, $CuCl_2.2H_2O$ and Cu $(NO_3)_2.3H_2O$ is dissolved in deionized water with a molar ratio of 1:1. The solution is stirred for about 30 min with a magnetic stirrer at room temperature. Then, 0.1 M of NaOH solution is added drop wise into the above prepared solution until pH reaches 14 under constant stirring. The pH is chosen to be 14 in order to obtain supersaturation. A black precipitate is obtained and the solution is filtered and washed several times using ethanol and deionized water up to pH 7. Followed this, precipitates are collected and dried overnight at 120 °C. To enhance crystallinity and obtain high crystalline CuO nanoparticles, it is further annealed at 500 °C for 4 h.

Characterization of CuO nanoparticles

Crystal structure, crystallite size, and crystallinity of synthesized samples are characterized by XRD, using a monochromatic beam of Cu Kα radiation (λ = 1.5406 Å) in an X'Pert Pro PANalytical X-ray diffractometer. An accelerating voltage of 40 kV and a current of 30 mA with a scan step time of 3.175 s are used. The XRD pattern is recorded in a 2θ range of 10 to 80° verses intensity with a step size of 0.0170°. The values of 2θ, d-spacing, relative intensity, and FWHM are measured from the XRD pattern. By comparing the X-ray peaks, d-spacing, and intensities obtained from the experiment with the JCPDS database, the crystal structure is identified. The lattice constants are determined using UNITCELL software. Average crystallite size for the sample is calculated using Debye-Scherrer's formula. Optical properties of CuO nanoparticles are determined by diffuse reflectance UV-Visible Spectrophotometer (DRS UV-Vis, Shimadzu UV-2700). Furthermore, vibrational and morphological studies of CuO are analyzed by characterization techniques such as FTIR and SEM (Perkin Elmer spectrum 2 and JOEL model SEM EV018).

Results and discussion

Structural studies

XRD pattern of CuO nanoparticles prepared by precipitation method is shown in **Figure 1**. The peaks in the XRD pattern are sharp, which indicates the nanocrystalline nature of the sample. Various intensities of the diffraction peaks arising from the planes, such as (1 1 0), (−1 1 1), (2 0 0), (−1 1 2), (−2 0 2), (1 1 2), (0 2 0), (2 0 2), (−1 1 3), (−3 1 1), (2 2 0), (3 1 1), and (0 0 4) confirm the formation of CuO nanoparticles. No other peaks are observed, which shows the purity of the sample. The observed 2θ values are compared with the standard data. The entire peaks match quite well with the JCPDS Card No: 80-1268, and absolutely recognize the monoclinic structure of CuO nanoparticles. The intensity of the diffraction peak at the (−1 1 1) plane is stronger, and reveals that more crystallites are oriented along the [-1 1 1] direction. The average crystallite size of CuO nanoparticles obtained by using Debye–Scherrer formula ($d = 0.9\lambda/\beta\cos\theta$., where λ is the wavelength of X-ray radiation, and β is the full width at half maximum (FWHM) of the peaks at the diffracting angle θ.) is found to be 28.82 nm, which indicates the particles have less value of strain [18]. The lattice parameters are evaluated using UNITCELL software, and the lattice constants and cell volumes are given in **Table 1** for comparison. This value is compared with standard value, and shows only slight variation in that value. Effect of temperature on CuO nanoparticles has great impact on crystallite size and lattice parameter. Increase in crystallite size and lattice parameter is due to atomic diffusion. Srivastava *et al.* [38] reported that, at high temperature, crystallite size becomes large.

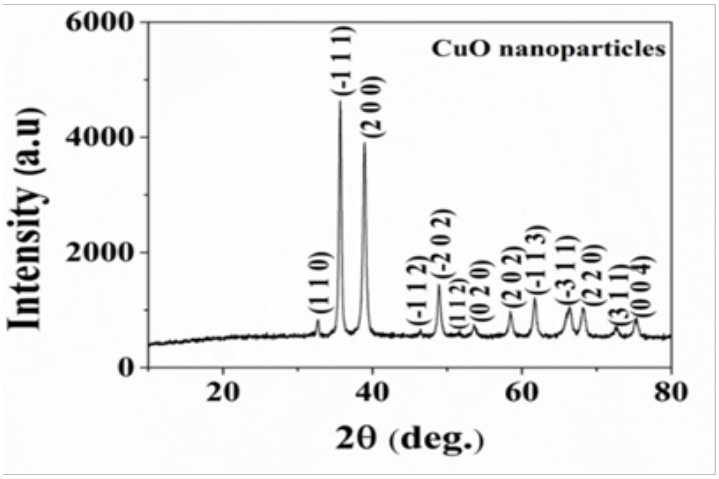

Figure 1 XRD pattern of CuO nanoparticles.

Table 1 Comparison of lattice constants and cell volume of CuO nanoparticles.

Lattice constants	Standard value	Observed value
a (Å)	4.6837	4.6833 (4)
b (Å)	3.4226	3.4208 (9)
c (Å)	5.1288	5.1294 (5)
β (deg.)	99.54	99.56 (2)
Cell volume $(Å)^3$	81.08	81.00 (3)

Optical studies

The reflectance spectra of the synthesized CuO nanoparticles are shown in **Figure 2**. The band gap for the sample is determined from the reflectance spectra using Kubelka-Munk (K-M) function. The optical band gap is calculated using Tauc relation $(\alpha h\nu)^2 = A(h\nu - E_g)$, where A - constant (depends on the transition probability), E_g - Optical band gap, h - Planck constant, and ν - Frequency. The incidental photon energy depends on the type of electronic transition. In this transition, the electronic momentum is conserved, and the transition is direct, but if the momentum is not conserved, the transition must be indirect. The E_g (eV) can be estimated by extrapolating a straight line to the $(\alpha h\nu)^2 = 0$ axis [36].

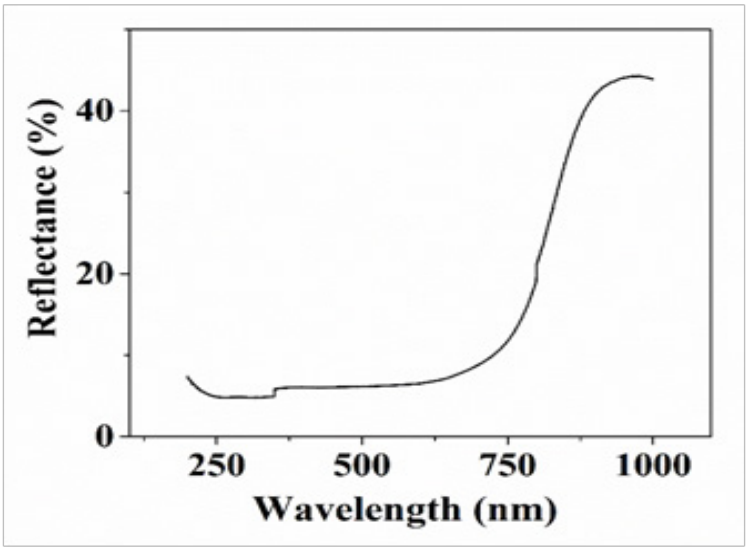

Figure 2 Diffused reflectance spectra of CuO nanoparticles.

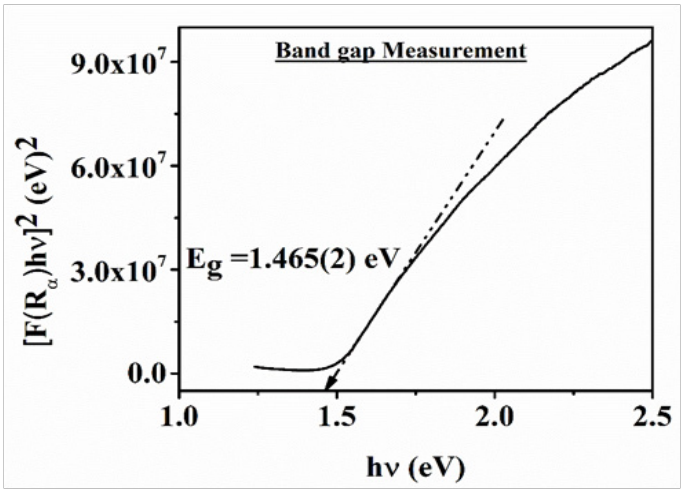

Figure 3 Tauc's plot of direct transitions of CuO nanoparticles.

Tauc's plot corresponds to measurement of band gap of the respective sample, which is shown in **Figure 3**. The value of the direct and indirect band gap is determined from the intercept of the straight line, which is found to be 1.4 and 1.6 eV, respectively. The optimum direct band gap is 1.465(2) eV; the optical properties of CuO make it a very suitable semiconducting absorber material for solar cell applications.

FTIR spectra

FTIR spectroscopy is a useful tool to identify functional groups in a molecule, because each specific chemical bond often has a unique energy absorption band; it can be used to obtain structural and bond information of a complex to study the strength and type of bonding. The FTIR spectra of synthesized CuO nanoparticles are recorded by KBr pellet method [37,38]. FTIR spectra of CuO NPs are depicted as wavenumber versus transmittance in **Figure 4**; active peaks are noticed in the range of 400 to 600 cm^{-1}, corresponding to stretching vibrations of CuO nanoparticles in the monoclinic CuO [19]. This implies that the peaks at 508 and 606 cm^{-1} are assigned for Cu-O stretching vibration [39,40]. The peak broadening of the absorption band represents that the CuO are in nanosize. As the size of as-prepared CuO nanoparticles is much less than the bulk form CuO, it has an IR peak of Cu-O stretching vibration and is shifted to the blue direction. The FTIR absorption of CuO nanoparticles is blue-shifted compared to that of the bulk form due to their quantum size effect and spherical nanostructures. The band at 1120 cm^{-1} represents triply degenerative ν_3 mode of SO_4^{-2} ions [10]. The peak appearing at 1417 cm^{-1} corresponds to C-O stretching of the carboxylate ion bond in the CuO nanoparticles [8]. The peak at 1567 cm^{-1} is ascribed to the water H-O-H scissoring vibration and carboxylate anion asymmetrical stretching [41]. The broad and strong absorption peak at 3415 cm^{-1} is associated with the absorbed water molecules by the nanoparticles from moisture [5,39,42]. The peak at 3478 cm^{-1} represents N-H vibration of amine group. The peak at 3555 cm^{-1} assigns a strong and broad O-H stretching intermolecular bond [40]. It supports the XRD results.

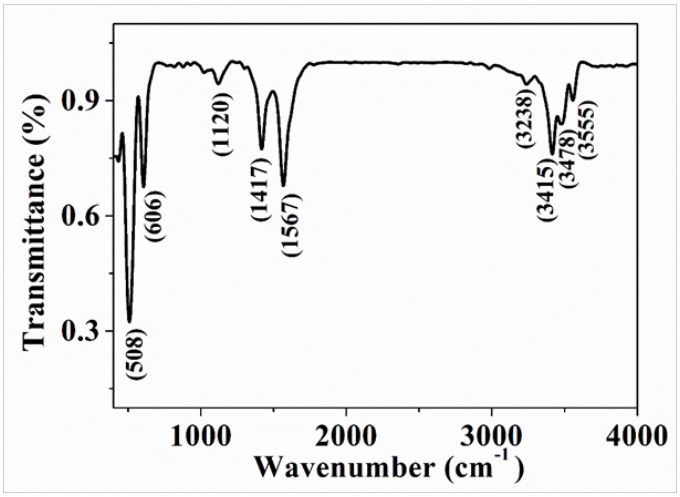

Figure 4 FTIR spectra of CuO nanoparticles.

Morphology studies

Figure 5 shows SEM images of the CuO nanoparticles at multiple magnifications. SEM analysis provides information on the size and morphology of CuO nanoparticles and their state of agglomeration. It can be seen from **Figure 5** that the CuO nanoparticles have spherical shapes, with some voids, and are well-dispersed with smooth surfaces. In addition, some flake-like particles are seen on surface **Figure 5(d)**. These few small particles aggregate into primary spherical particles because of their extremely small dimensions and high surface energy.

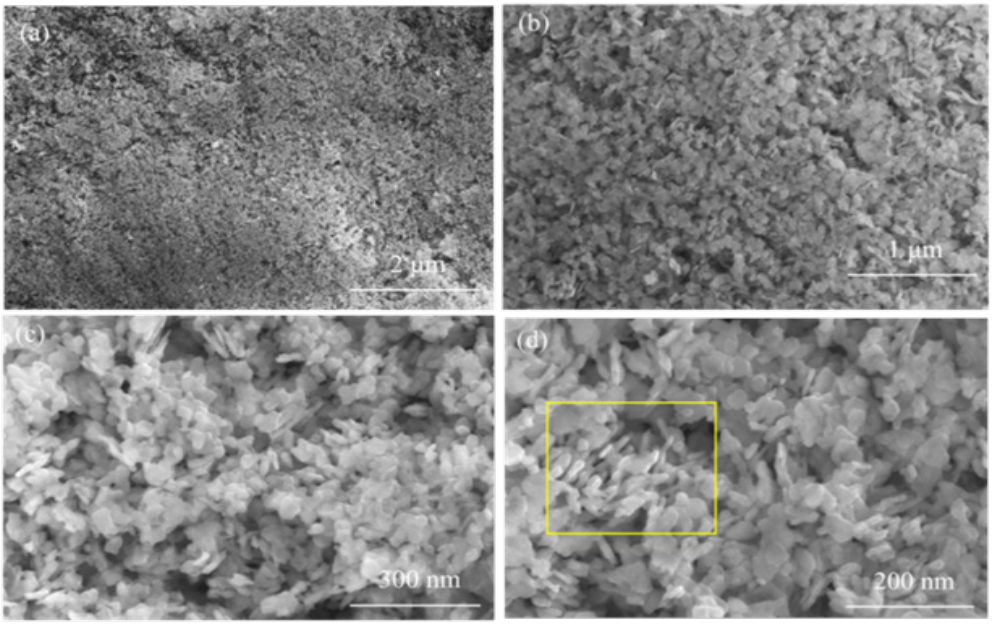

Figure 5 SEM images of CuO nanoparticles at different magnifications (a) 2 μm, (b) 1 μm, (c) 300 nm, (d) 200 nm.

Therefore, CuO nanostructures in the large area are a positive step for the development of low-cost and temperature fabrication solar cells.

Conclusions

In this work, CuO nanoparticles are successfully prepared by precipitation method. Characteristic studies, such as XRD, UV-DRS, FTIR, and SEM, are utilized for the analyses of structural, optical, vibrational, and morphological properties. Crystallite size of synthesized nanoparticles is 28.82 nm and has a monoclinic crystal structure. The observed direct band gap is 1.465(2) eV, which is much smaller than that of bulk CuO, which ascertains its suitable material capability for solar cells. FTIR spectra also validates the purity of CuO nanoparticles and the formation of nanosized particles through blue shift. For a single P-N junction solar cell, the optimum bandgap is 1.4 eV, which has been achieved in our work. This optical property of CuO and good electron mobility make it a suitable solar cell absorber material for solar cell application. In future, it will be synthesised by green synthesis method, and be used for the investigation of enhanced solar cell efficiency. Moreover, efficiency of this material will be strengthened by the inclusion of carbon.

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