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Synthesis And Characterization of Copper Oxide Nanoflakes By Co-Precipitation Method

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ABSTRACT

Copper oxide (CuO) nano flakes were successfully prepared by a co-precipitation method. X-Ray diffraction patterns confirm that the films have polycrystalline nature with monoclinic structure. The SEM analysis revealed that golf ball structure with randomly oriented surface feature was observed for the CuO nanoparticles deposited at the high substrate temperature. The EDS analysis showed the presence of Cu and O in the films. In summary, various results indicate that this technique is a low temperature, cheap, and fast method for the producing CuO nanostructures. The average particle size was 21 – 34 nm. The crystallite size CuO nanoparticles of the observed from the SEM data matched with the estimates obtained from the XRD data. The results obtained indicated that the co-precipitation method is a promising low temperature, cheap and fast method towards the preparation of CuO nanostructures.

Keywords: CuO_2 nanoparticles; TEM; Co-precipitation method; Morphology

INTRODUCTION

In recent years, the research on semiconductor metal oxide nanostructures have been the subject of focused research, owing to their unique electronic, optical, mechanical, magnetic and chemical properties. Chiefly, the research on copper oxide (CuO) nanostructures has been elicited intensely due to its facile preparation of peculiar morphologies, such as nanorods, nanoflowers, nanowires, nano dendrites, nanosheets, etc. In terms physical and chemical properties, CuO is a white solid inorganic powder, non-flammable, stable, insoluble in water, II-VI semiconductor with a wide band gap energy of 3.3 eV and high excitation energy (60 eV) [1]. Besides, CuO has several favorable properties, viz. good transparency, high electron mobility, low toxicity, photo chemical stability and higher breakdown field strength.

CuO is usually exists in two crystalline forms, hexagonal wurtzite and cubic zinc blende, among them the wurtzite structure (B4 type) can only be obtained at optimum pressure and temperature [2-3]. In the typical wurtzite hexagonal structure of CuO, oxygen (O) and copper (Cu) atoms are spatially arranged in a manner that O atoms are arranged in a closed hexagonal structure, while the Cu atoms occupy the center of the distorted tetrahedron structure [4]. The variety of structures of nano-metric CuO means that they can be classified among new materials with potential applications in several fields of nanotechnology. CuO can occur in one, two and three dimensional nanostructures. Among them, one dimensional nanostructure make up the largest group, including nanorods, nanoneedles, nanohelixes, nanosprings, nanorings, nanoribbons, nanotubes, nanobelts, nanowires, nanocombs, etc. The examples for two dimensional CuO nanostructures include nanoplates, nanosheets, nanopellets, etc.

One of the most important environmental applications of nanotechnology is in the water sector. Heterogeneous photocatalysts, one of the advanced oxidation process (AOPS), is a cost-effective treatment method towards the removal of toxic pollutants in industrial waste water disclosing its ability to convert these into safer products, such as CO_2, H_2O and mineral acids [5-11]. Several conventional methods have been adopted for synthesis of ZnO nanostructures, viz. chemical vapour deposition [12-18], laser ablation [19-23], solvothermal [24-25], thermal decomposition [25-26] and sol-gel method [26-27]. In the present work, we introduce a simple co-precipitation method to synthesize uniform and pristine CuO nanoflakes using copper nitrate as a metal precursor and ammonium hydroxide as a precipitating agent. The resultant

nanostructured materials are characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), selected area electron diffraction (SAED), Fourier transform infrared spectroscopy (FTIR) and energy dispersive X-Ray spectroscopy (EDS) characterization techniques and the results are discussed in detail.

EXPERIMENTAL PROCEDURE

MATERIALS

Copper nitrate ($Cu(NO_3)_2.6H_2O$), and ammonia solution of analytical grade were used as such without further purification for synthesis process. Double distilled water was used throught the experiments.

SYNTHESIS OF CUO NANOPOWDERS

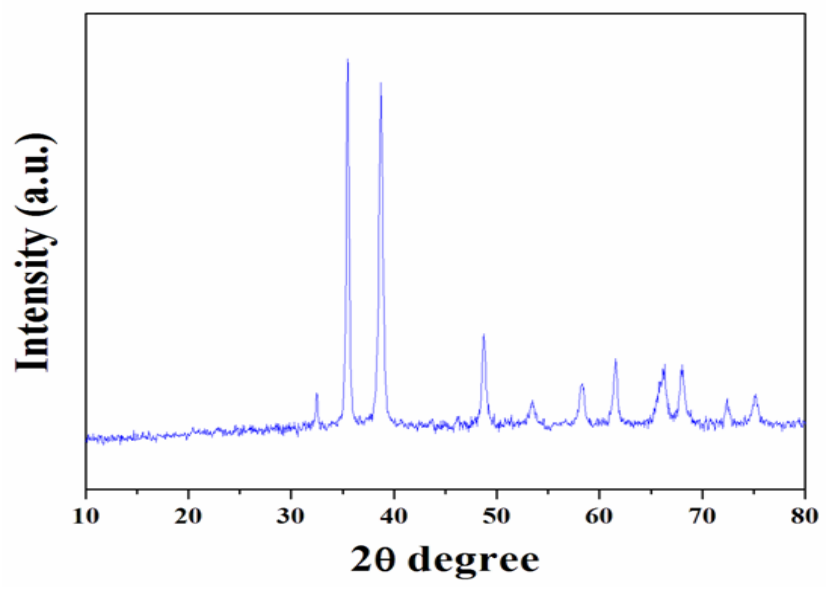
The synthesis of CUO nanoparticles by powder method was carried out as follows. First, a 0.1 M of Cu hydroxyl solution was prepared by dissolving Cu nitrate with in deionized water. Then pH of the solution was maintained at 8 by adding liquid ammonia solution drop wise. The resulting product was filtered and washed with double distilled water and ethanol until it became free from impurities. The precipitate was irradiated for 5 minutes in household microwave (radiation frequency 2.45GHZ, Power up to 1 KW) with convection mode, giving a white product. Finally the sample was as prepared by annealed temperature at 400 °C for 4 hours.

CHARACTERIZATION STUDIES

The crystallinity of the prepared CuO nanoparticles were examined by X-ray diffraction (XRD) using a Bruker AXS D8 Advance diffractometer with monochromatic CuKα wavelength of 1.5406 Å. The samples morphology was observed by scanning electron microscopy (SEM) using a JEOL 5600LV microscope at an accelerating voltage of 10 kV, transmission electron microscopy (TEM) and selected-area electron diffraction (SAED) in a Tecnai G20-stwin operated at 200 kV. The Fourier transform infrared spectra (FT-IR) of the samples were recorded using a Nicolet 5DX FTIR spectrophotometer.

RESULTS AND DISCUSSION

X-Ray Diffraction Analysis



X-ray diffraction patterns of CuO nanoparticles annealed at 400^{o}C.

The X-ray diffraction patterns of CuO nanoparticles prepared at substrate annealed at 600^{o}C are shown in Fig. 1. The position of the diffraction peaks observed in all CuO sample match well with those reported for polycrystalline nature with monoclinic structure. The X-ray diffraction reveals that all films are having monoclinic structure with XRD peaks correspond to (11 0), (1 1 1), (1 1 2), (02 1), (311) and (3 1 1) planes. The different peaks in the diffractogram were indexed

and the corresponding values of inter planar spacing ''d'' were calculated and compared with standard value (JCPDS data (80-1916)). The average crystallite size calculated from the XRD data using Scherrer formula,

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

Where λ is the wavelength of X-ray (λ = 1.542Å) (CuKα), (h k l) are the Miller indices, β is the full width at half maximum (FWHM) of the line, and θ is the diffraction angle. In addition to the increase in the intensity of the diffraction peaks with increase doping concentration which is depicted the increase of crystallinity. The average crystalline size is 26 nm.

SCANNING ELECTRON MICROSCOPE (SEM)

Fig. 2(a–d) shows the scanning electron micrographs of CuO nanoparticles deposited at 400^{o}C. But the optimum substrate temperature (400^{o}C), the particles were found to be golf ball structure and possess distinct grain boundaries. But the morphology was changed into uniform golf ball structure at CuO nanoparticles. The crystallite size CuO nanoparticles of the observed from the SEM data matched with the estimates obtained from the XRD data.

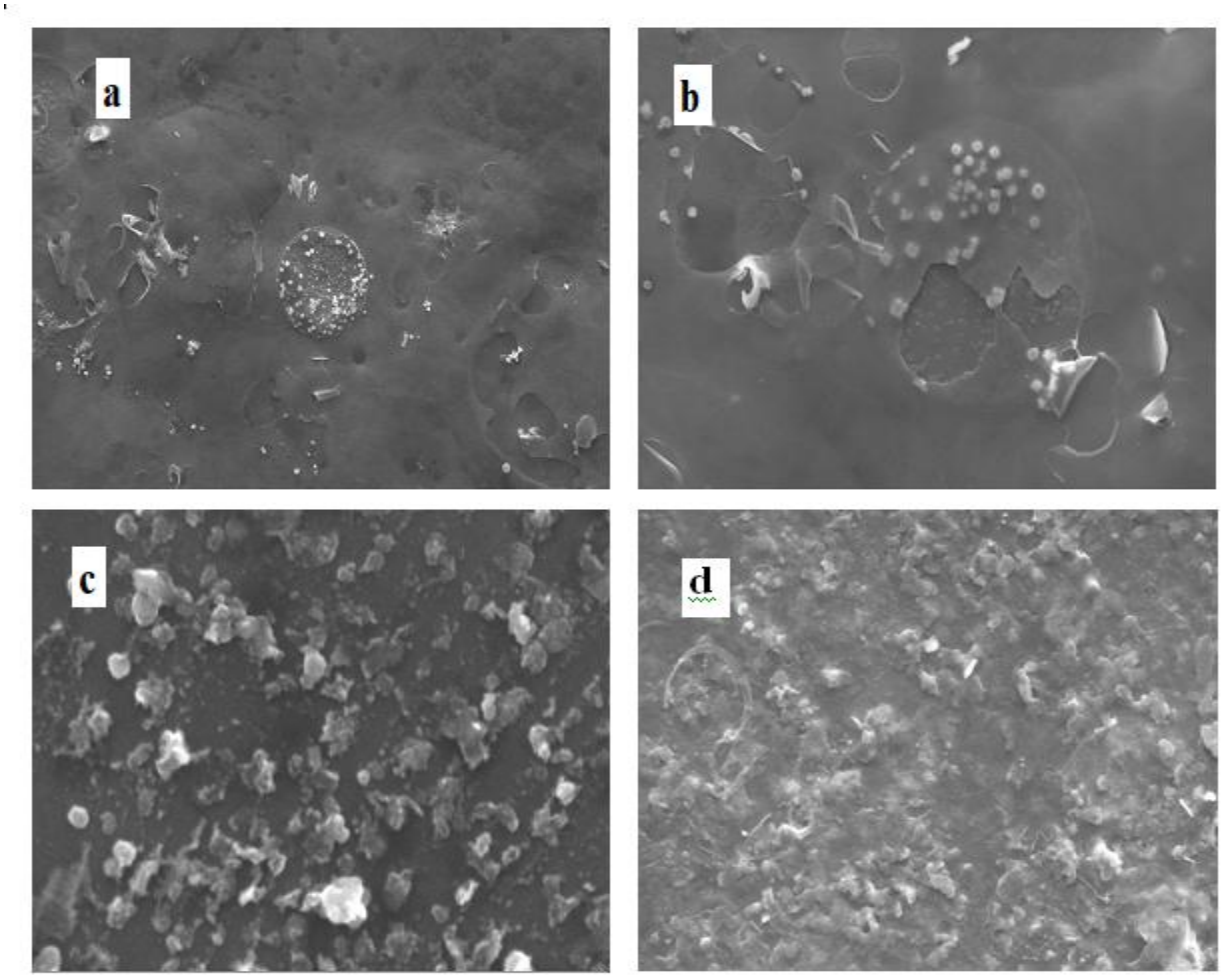
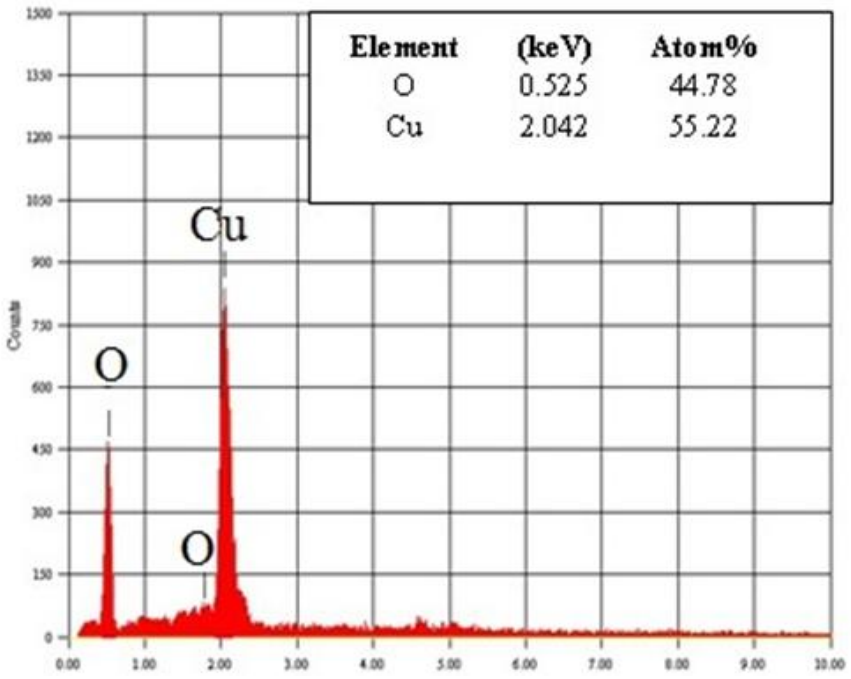


Fig. 2 (a-d). Scanning electron microscope image of CuO nanoparticles annealed at 400^{o}C.

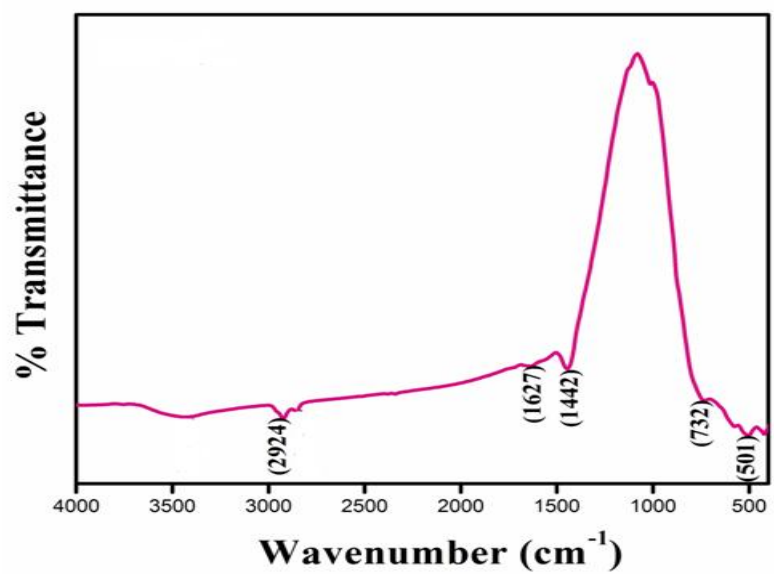
ENERGY DISPERSIVE SPECTRUM (EDS) ANALYSIS

Energy Dispersive Spectral (EDS) analyses have been used to identify the elements present in sample. The weight percentage (Wt %) were measured and these values are shown in the corresponding spectra. There is no any evidence for the presence of other impurities in the fabricated samples. Finally, EDS report is presence of Cu and O only.



Energy dispersive spectrum of CuO nanoparticles annealed at 400°C.

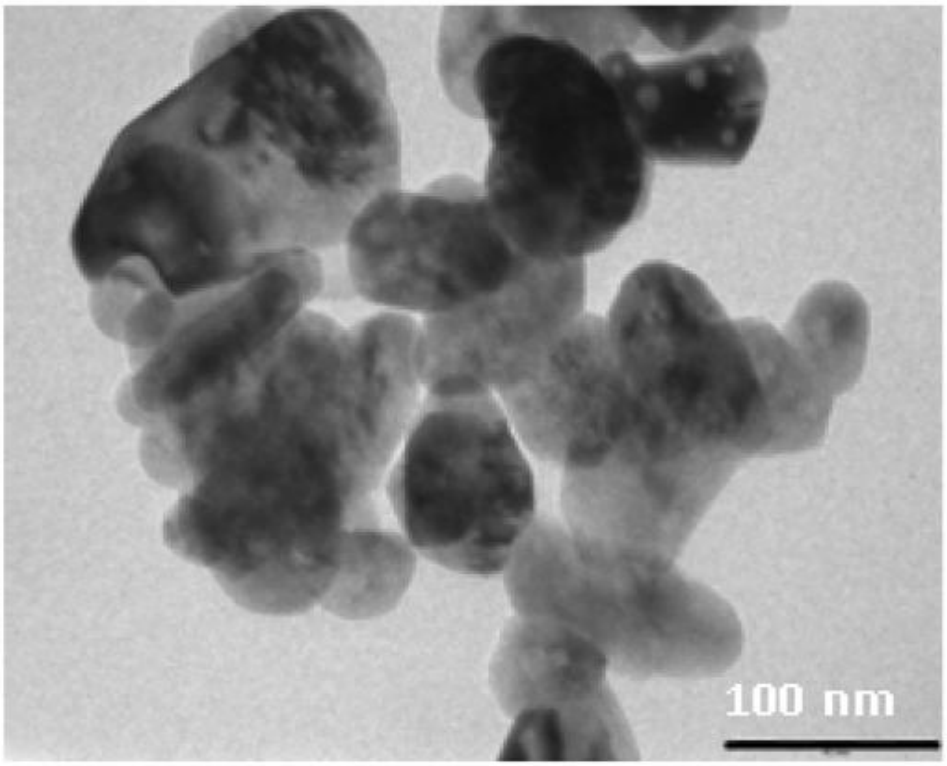
Fourier Transform Infra-red Spectroscopy (FTIR)



Fourier transforms infra-red spectroscopy of CuO nanoparticles annealed at 400°C.

The IR spectroscopy was used to learn the surface interactions of the adsorbed water in dynamic symmetry with the gas phase on the CuO surface. Frequency shifts and absorbance values were carefully observed to interpret the surface structure of the CuO phase. It is well-known that H_2O and CO_2 molecules are simply chemisorbed onto the CuO surface as soon as exposed to the ambiance.

TRANSMISSION ELECTRON MICROSCOPE (TEM)



TEM IMAGE WAS CUO NANOPARTICLES ANNEALED AT 400^{O}C.

The TEM image particle was found to be golf ball structure and possess distinct grain boundaries. But the morphology was changed into uniform golf ball structure at CuO nanoparticles. The crystallite size CuO nanoparticles of the observed from the SEM data matched with the estimates obtained from the XRD data. The average particle size was 21 – 34 nm.

CONCLUSION

Pristine CuO were prepared by a facile co-precipitation method. X-Ray diffraction patterns confirm that the films have polycrystalline nature with monoclinic structure. The SEM analysis revealed that golf ball structure with randomly oriented surface feature was observed for the CuO nanoparticles deposited at the high substrate temperature. The EDS analysis showed the presence of Cu and O in the films. In summary, various results indicate that this technique is a low temperature, cheap, and fast method for the producing CuO nanostructures. The average particle size was 21 – 34 nm. The crystallite size CuO nanoparticles of the observed from the SEM data matched with the estimates obtained from the XRD data. The fabricated CuO nanoparticles are promising semiconductor materials for optoelectronic applications. The study suggests that the co- precipitation is a simple and cost effective approach towards the synthesis of CuO nanostructures.

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