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Synthesis and Characterization of Copper Oxide (CuO) Nanoparticles by Solution Combustion Method

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ABSTRACT:- *In the last decennium, the progress of nano size materials of metal and metal oxide particles are effectively prevailing due to their protuberance in diverse fields of applications. Among the oxides of transition metals, Monoclinic copper oxide nanoparticles are of special interest because of their efficiency as nano fluids in heat transfer application, captivating size-dependent chemical properties and optical properties. In the self-combustion technique, nanoparticles were obtained by heating the materials until the mixture combusts at 200°C. CuO nanoparticles were synthesized from Cu $(NO_3)_2.3H_2O$ as oxidizer and glycine as fuel. The material was stirred at 250 r.p.m continuously for 45 minutes. The mixture was then heated up until it combusted at 200°C. Samples were then annealed at 800°C for 1 hour. Relevant properties of as-synthesized and annealed nanoparticles were investigated by X-ray diffraction (XRD), scanning electron microscope (SEM) and Fourier Transform Infrared (FTIR) Spectroscopy. Overall results suggest that the formation of CuO nanostructures with different shape, size and morphology can be achieved using a particular fuel and oxidizer ratio. The improvement in their crystallinity and purification can be further attained by post calcination process.*

Keywords: *CuO nanoparticles, Solution combustion method, Glycine.*

INTRODUCTION

Nanoparticles with individualistic properties have emanated as an interdisciplinary field comprising multitudinous fields of science. Metal oxide nanoparticles have manifested a great interest in field of optoelectronics, catalysis and solar cells due to their notable physical and chemical properties differing from bulk. The wide ranges of properties exhibited by the metal oxide nanoparticles have enticed enormous researchers to analyze on their heterogeneous catalysis applications [1]. Apart from all the metal oxides, the Copper oxide nano particles have been the center of attraction for the past few denary. This is due to the presence of elevated intensifying properties such as the high chemical reactivity due to their overwhelming surface to volume ratio. In general the copper oxide nano particles are of monoclinic structure and exhibits p-type semiconductor behavior with an indirect band gap of 1.21-1.51eV [2].These nanoparticles have a wide range of applications in divergent fields. There have thus far been numerous methods developed for the preparation of copper oxide nanoparticles, including spray pyrolysis, alcohol thermal, sonochemical, sol-gel, solvothermal, chemical vapour deposition method, solution combustion and hydrothermal method. All of these methods require expensive precursors and time-consuming.However, among the methods reported in the literature, solution combustion (SC) has proved one of the more successful methods for the synthesis of metal oxide nanoparticles and this method is environmentally benign. In this study, pure Copper oxide nanoparticles were prepared by solution combustion synthesis and their structural and the morphological properties were studied.

EXPERIMENTAL

The solution combustion technique was used in the synthesis of Pure CuO nanoparticles with the help of high graded chemicals. The oxidizer and fuel are the two elements in the combustion synthesis where, the oxidizer proffers oxygen for the combustion reaction and the fuel is a substance that aids in

burning. When the oxidizer and the fuel are mixed in an appropriate proportion an exothermic chemical reaction takes place [3]. Stoichiometric composition of the redox mixture was calculated based on the principles of propellant chemistry, keeping the O/F ratio to unity. Copper nitrate tri hydrate Cu $(NO_3)_2.3H_2O$ was used as an oxidizer and glycine as a fuel and their required amounts were dissolved in distilled water. The homogeneous mixture was then kept on hot plate to evaporate the excess water .It was then allowed to auto-ignite with the rapid evolution of large volume of gases to produce fine powder. Voluminous and porous nanocrystalline black-colored product was obtained. The As-prepared powder was heat treated at 800 ^{0}C for 1 hr to remove any carboneous impurity present in it and hence pure and well crystalline powder was obtained. The chemical reaction is given by the following equation:

$Cu(No_3)_2 + C_2H_5NO_2 \rightarrow CuO + H_2O + N_2 + CO_2$ ----- (1)

$9Cu(No_3)_2 + 10C_2H_5NO_2 \rightarrow 9CuO + 25H_2O + 14N_2 + 20CO_2$ ----- (2)

The sample is allowed to cool at room temperature and investigated by X-Ray Diffractometer (XRD). The morphology was monitored by scanning electron microscope (SEM).Chemical properties were investigated by Fourier transform infrared spectroscopy [4].

RESULTS AND DISCUSSIONS

Structural studies of these samples were carried out by XRD (Phillips-3710 powder X-ray diffractometer Cu $K\alpha_1$ radiation of wavelength 1.54056Å). The XRD pattern was compared with standard JCPDS files of CuO (89-5895). The morphology of the powder was studied using scanning electron microscopy (SEM) (JEOLJSM- 6360). The FTIR absorption behavior of these samples was obtained using Perkin-Elmer FTIR spectrometer with scanning range 4000 cm^{-1}–400 cm^{-1}.

Structural analysis: X-ray diffraction

The XRD pattern of the synthesized and annealed CuO nanoparticles is shown in Fig. 1. It reveals a monoclinic structure with Lattice parameters *a* = 4.461Å,b = 3.125Å, *c* = 5.058Å. The intensities and positions of peaks are in accordance with that of the standard spectrum (JCPDS no. 89-5895). In addition no impurity peaks where observed in the XRD pattern. This result exhibits that the combustion synthesis has emanated in high purity CuO nanoparticles.
The Crystallite size was calculated and determined using the Scherrer Formula

$$D = \frac{0.9\,\lambda}{\beta \cos\theta} \quad \text{----- (3)}$$

where λ is the wavelength of X-ray radiation, β is the full width at half maximum (FWHM) of the peaks at the diffracting angle θ. Crystallite size was calculated for pure copper oxide from equation (3)and reported as 32 nm[5] .

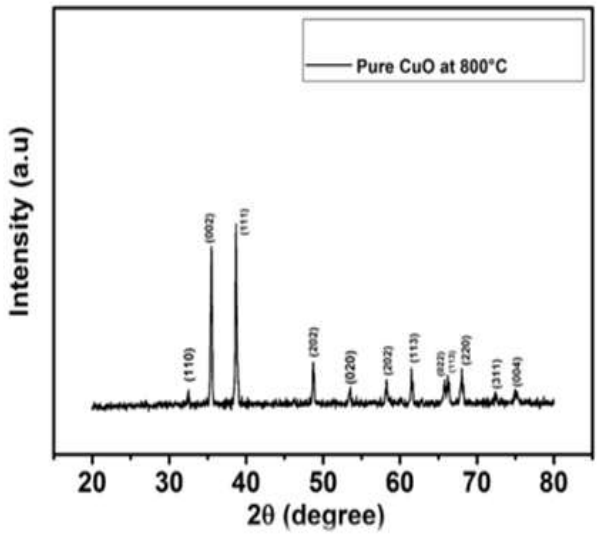


Figure1. XRD pattern of Pure CuO nano particles

The crystallite size and the lattice parameter values for pure CuO annealed at 800°C are given in Table 1.

Table 1. Estimated lattice parameters and crystallite size

Crystallite size	a (Å)	b (Å)	c (Å)	D(nm)
Pure CuO	4.461	3.125	5.058	32

Morphological Study: SEM

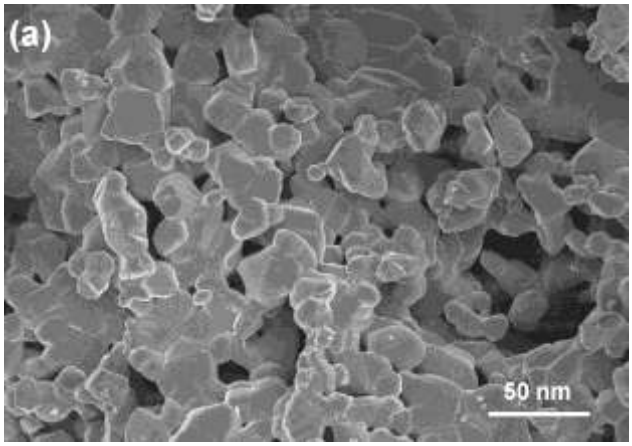
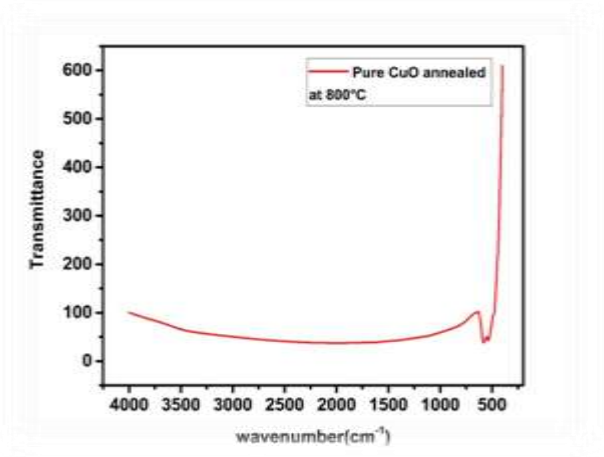


Figure2. SEM micrograph of Pure CuO

The Figure 2 shows the Scanning Electron micrograph of the CuO nanoparticles annealed at 800°C. In combustion, appreciable proportion of gases was evolved during reaction which consequently gave a loose and porous structure. Conventionally, combustion reactions produce homogenous material of small and uniform nanoparticles, but because of high temperature provoked during the reaction it results into agglomeration. This implies that the powder appears spongy where the nanoparticles are linked together to form agglomerates of different sizes and shapes contributing various facets. The average particle size from the SEM images was found to be around 30-40 nm. From the SEM images, it is perceived that pores and voids in the image are due to the escape of gases during the combustion reaction.

FTIR Analysis:

Figure3. FTIR Spectra of Pure CuO



The FTIR spectra of a pure metal doped CuO nanocrystallites derived by combustion method was documented at room temperature in the range 400 cm^{-1} - 4000 cm^{-1} is shown in Fig.3.The Spectra shows a palpable transmittance band around 580 cm^{-1} and 535 cm^{-1} which are consigned to vibration of Cu(II)-O bands stretching mode[6]. The broad absorption band at 1712 cm^{-1} is due to the vibration mode of engrossed water molecules.

CONCLUSION:

In abridgement, CuO nanostructures were successfully synthesized by solution combustion using copper nitrate tri hydrate (Cu $(NO_3)_2$).$3H_2O$ as an oxidizer and glycine as fuel. It is inferred that annealing process can effectively remove residue and lead to the better crystallization of CuO nanoparticles. X-ray diffraction results suggest the formation of monoclinic CuO nanoparticles and the Crystallite size was found to be 32 nm. From SEM study, it was inferred that nanoparticles are linked together in agglomerates of different sizes and shapes with average size of 40 nm. FTIR results suggest Cu-O stretching modes. Furthermore, it is notified that a definite fuel oxidant ratio has strong influence on shape, size and morphology of CuO nanostructures.

Acknowledgement:

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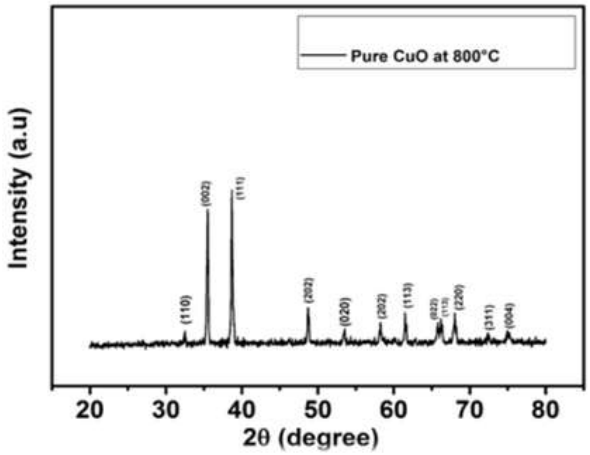

Figure1. XRD pattern of Pure CuO

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Figure 1

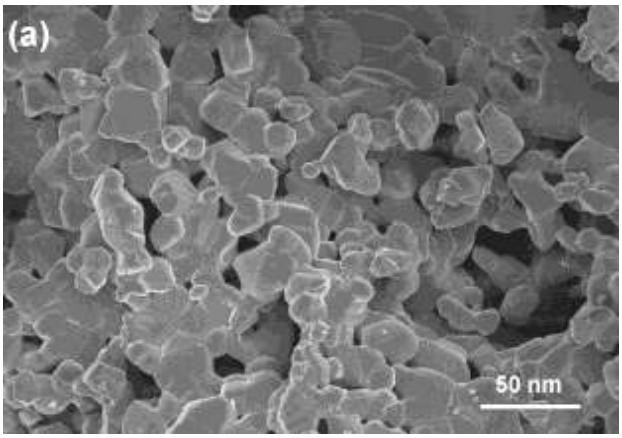

Figure2. SEM micrograph of Pure CuO

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Figure 2

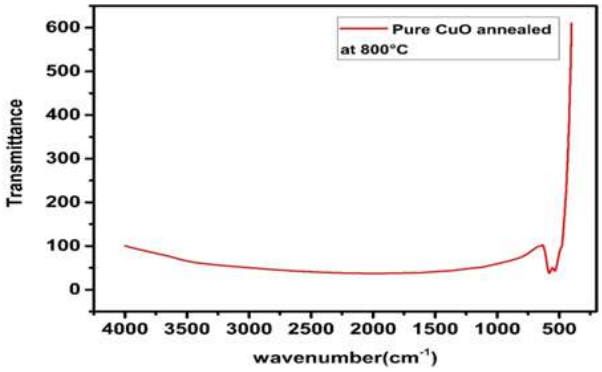

Figure3. FTIR Spectra of Pure CuO

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Figure 3

Table 1. Estimated lattice parameters and crystallite size

Crystallite size	a (Å)	b (Å)	c (Å)	D(nm)
Pure CuO	4.461	3.125	5.058	32

Table 1

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