



International Journal of Advance Engineering and Research Development

National Conference On Nanomaterials, (NCN-2017)

Volume 4, Special Issue 6, Dec.-2017 (UGC Approved)

SYNTHESIS AND CHARACTERIZATION OF COPPER OXIDE NANOPARTICLES

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Copper Oxide nanoparticles have been synthesized by co-precipitation method and the synthesized Copper Oxide nanoparticles were annealed at 500^oC. Crystalline nature of the synthesized Copper Oxide Nanoparticles was confirmed from X-Ray Diffraction analysis. The SEM images of copper oxide nanoparticles particles are rod like structure and slightly agglomerated. The Photoluminescence spectra of Copper Oxide nanoparticles annealed at 500^oC revealed that two emission peaks are observed at 420 and 440nm. Therefore the synthesized Copper Oxide nanoparticles by co-precipitation method are very promising and may have extensive applications.

Keywords: *co-precipitation, nanoparticles, annealing, photoluminescence.*

1. Introduction

Metal oxide nanoparticles have shown their great interest in field of sensing optoelectronics, catalysis and solar cells due to their unique physical and chemical properties differing from bulk. Among all the metal oxides copper oxide nanomaterials have attracted more attention due to its unique properties. Copper oxide nanomaterials have the advantage of a lower surface potential barrier than that of metals, which affects electron field emission properties. Copper oxide is considered as a potential field emitter, an efficient catalytic agent, as well as a good gas sensing material. It also plays an important role in optoelectronics and solar cell¹⁻⁷.

Due to the potentiality of CuO, it acts as a catalyst; whereas all metal oxides are not useful for the catalytic activity. In the fabrication of super capacitors also CuO is very useful and in nano range it has the wide band gap nearly equal to ZnO. The favourable band gap of CuO around 2.6 eV makes it useful for solar energy conversion and it can be used as solar cell window material. Cu and Cu₂O nanoparticles have been synthesized through different methods such as thermal decomposition, metal salt reduction, microemulsion, laser ablation, DC arc discharge, solvothermal and sonochemical reactions⁸⁻¹². Among various techniques for synthesis, the co-precipitation method of producing metal oxide nanomaterials is unique and economical. Chemical coprecipitation can produce fine, high-purity, stoichiometric particles of single and multicomponent metal oxides. Furthermore, if process conditions such as solution pH, reaction temperature, stirring rate, solute concentration and surfactant concentration are carefully controlled, oxide particles of the desired shape and sizes can be produced¹³. The present work is focused on the cost-effective synthesis and characterization of nanometer-sized ultrafine particles with monodispersity, carried out via soft chemical approach with controlled properties.

2. Experimental Details

In the present work Copper oxide nanoparticles were synthesized by co-precipitation method. All chemicals used in the experiment were of analytic reagent grade and purchased from Merck, India.. For the synthesis of copper oxide nanoparticles copper chloride (CuCl_2) and copper nitrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) were used as a precursors. The precursor solution was stirred in a magnetic stirrer for 2 hours. 0.7133 grams of NaOH pellet were dissolved in 50ml deionized water. The prepared NaOH solution was added slowly in to the precursor solution till the pH value reached to 14. Then the complete solution was kept for aging about 12 hours. After 12 hours the black colored precipitate was formed. The black colored precipitate was taken out by filtering and then the filtered powder was washed with deionized water for 3 times Finally the powder washed with Ethanol to remove the unreacted by products. The washed powder was dried at 160°C for 9 hours by using hot air oven. After drying the collected copper oxide nano powder were annealed at 500°C for 1 hour by using Muffel furnace.

The powder X-ray diffraction (XRD) was performed using X-ray diffractometer system Philips PW 11/90, with nickel filtered $\text{CuK}\alpha$ ($\lambda = 1.5405 \text{ \AA}$) radiation. Photoluminescence (PL) measurements were performed by F-4500 FL spectrophotometer with 150 W xenon lamp at room temperature. Powder samples are spread over a glass slide and mounted inside the sample holder. The morphology of CuO nanoparticles was studied using scanning electron microscope (JEOL JSM 5600).

3. Results and Discussion

Structural Analysis (XRD)

Crystalline nature of the synthesized Copper Oxide Nanoparticles was confirmed from X-Ray Diffraction analysis. Fig.1a. shows the X-Ray Diffraction pattern of Copper Oxide Nanoparticles. All the diffraction peaks are well matched with monoclinic structure of copper oxide (JCPDS file No.048-1546). The characteristic peak located at $2\theta = 33.78^\circ$, 36.67° , 38.97° , 49.94° , 53.62° , 56.16° , 61.32° , 66.75° and 68.28° are assigned to (110), (002), (111), (202), (020), (202), (113), (311), (113) and (311) planes respectively. The grain size can be calculated using Debye - Scherer's formula from the full width half maximum (FWHM). Average grain size of the Copper Oxide Nanoparticles was calculated from Debye Scherer formula. The average grain size of copper oxide nanoparticles is found to be 11nm. Lattice parameters from XRD calculated that $a=4.53$, $b=3.41$, $c=5.12$. The calculated lattice parameters are well matched with the JCPDS values.

Fig.1b. shows the XRD pattern of copper oxide nanoparticles annealed at 500°C . The average grain size of copper oxide nanoparticles annealed at 500°C is found to be 14nm. After annealing process the peak intensity was increased. The characteristics peak located at $2\theta = 32.68^\circ$, 35.37° , 38.52° , 47.72° , 58.48° , 62.52° , 66.74° , 67.75° and 68.68° are assigned to (110), (002), (111), (202), (020), (202), (113), (311), (113) and (311) plane orientation of copper oxide (JCPDS file No 048-1546). This indicates the crystallinity of copper oxide nanoparticles was enhanced by the sufficient nucleation energy from annealing treatment^{14,15}. The lattice parameters of the copper oxide nanoparticles annealed at 500°C also well matched with the JCPDS value of copper oxide nanoparticles. The calculated grain size and the lattice parameters are

tabulated in table.1. From the XRD result it is inferred that the grain size increases when annealing the copper oxide nanoparticles as evidenced from the previous result.

SEM Analysis

Fig.2a represents the SEM images of copper oxide nanoparticles synthesized by co- precipitation method. It shows that the synthesized copper oxide nanoparticles formed have an irregular plane structure with no distinct morphology. Some particles are rod like structure and particles are slightly agglomerated. Fig.2b represents the SEM images of copper oxide nanoparticles annealed at 500⁰C. After annealing the copper oxide nanoparticles at 500⁰C the nanoparticles are found to be good dispersion and nano-flakes like structure. The agglomeration was reduced with increase in grain size.

Photoluminescence Analysis

Fig.3 shows the photoluminescence spectra of Copper Oxide nanoparticles annealed at 500⁰C. Two emission peaks are observed at 420nm and 440nm (blue) for copper oxide nanoparticles. The presence of emission bands in the lower wavelength region (420 & 440 nm) UV region may be attributed to Near Band Edge emission (NBE)¹⁶. The near band edge emission is attributed to the emission originated from the recombination of electrons and holes of copper oxide free excitons. Band edge emission is when an electron moves from the lowest energy excited state, to the highest energy rest state (from the bottom edge of the conduction band, to the top edge of the valance band). This energy difference is referred to as the band gap energy. When the electron drops into the valance band, it releases a photon of the exact same energy, the band gap energy. This process is band edge emission.

4. Conclusion

Copper Oxide nanoparticles have been synthesized by co-precipitation method and the synthesized Copper Oxide nanoparticles were annealed at 500⁰C. Crystalline nature of the synthesized Copper Oxide Nanoparticles was confirmed from X-Ray Diffraction analysis. The average crystalline size of copper oxide nanoparticles is found to be 11nm. The average crystalline size of copper oxide nanoparticles annealed at 500⁰C is found to be 14nm. The calcination temperature has more effect on the nanocrystallite growth and is in potential use foran advanced functional materials. The SEM images of copper oxide nanoparticles particles are rod like structure and slightly agglomerated. After annealing the copper oxide nanoparticles at 500⁰C the nanoparticles are good dispersion and so found to be nano-flakes like structure. The agglomeration was reduced with increase in grain size. The Photoluminescence spectra of Copper Oxide nanoparticles annealed at 500⁰C revealed that two emission peaks are observed at 420 and 440nm. This synthetic methodology is useful for fabricating efficient and cost-effective gas sensors and other allied potential applications.

Acknowledgments

The authors are grateful to the Secretary, Director, Principal and Head of the Department of Physics, Sri G.V.G Visalakshi College for Women, Udumalpet for their excellent encouragement and support.

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Figure captions

Figure 1a. XRD Pattern of Copper Oxide Nanoparticles

Figure 1b. XRD Pattern of Copper Oxide Nanoparticles Annealed at 500⁰C

Figure 2a. SEM images of Copper Oxide Nanoparticles.

Figure 2b. SEM images of copper oxide nanoparticles annealed at 500⁰C.

Figure 3. Photoluminescence spectrum of Copper oxide Nanoparticles annealed at 500⁰C

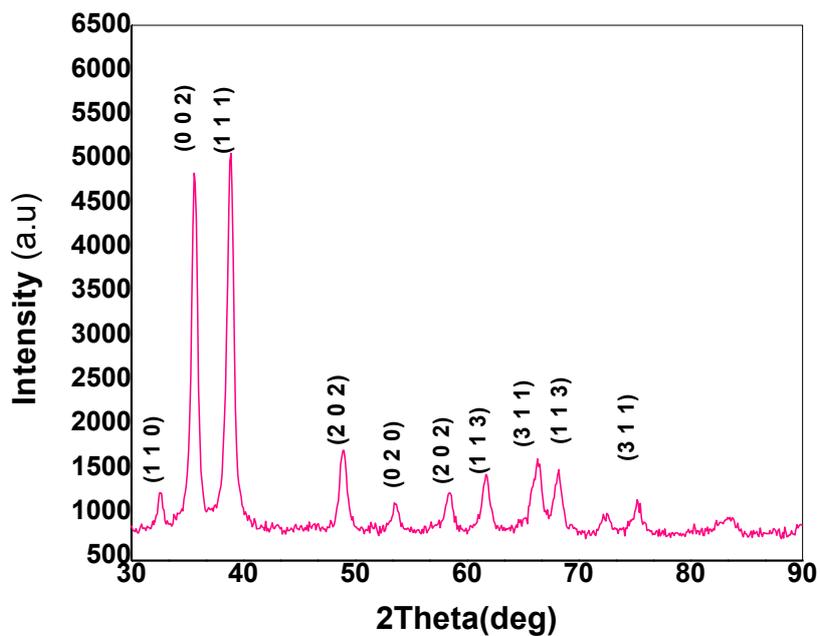


Figure 1a. Nirmala et al.

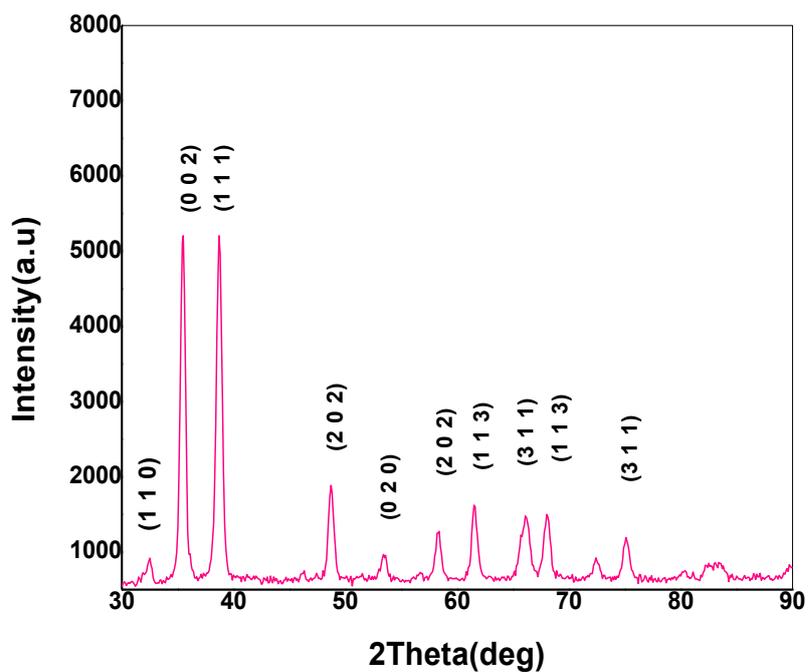


Figure 1b. Nirmala et al.

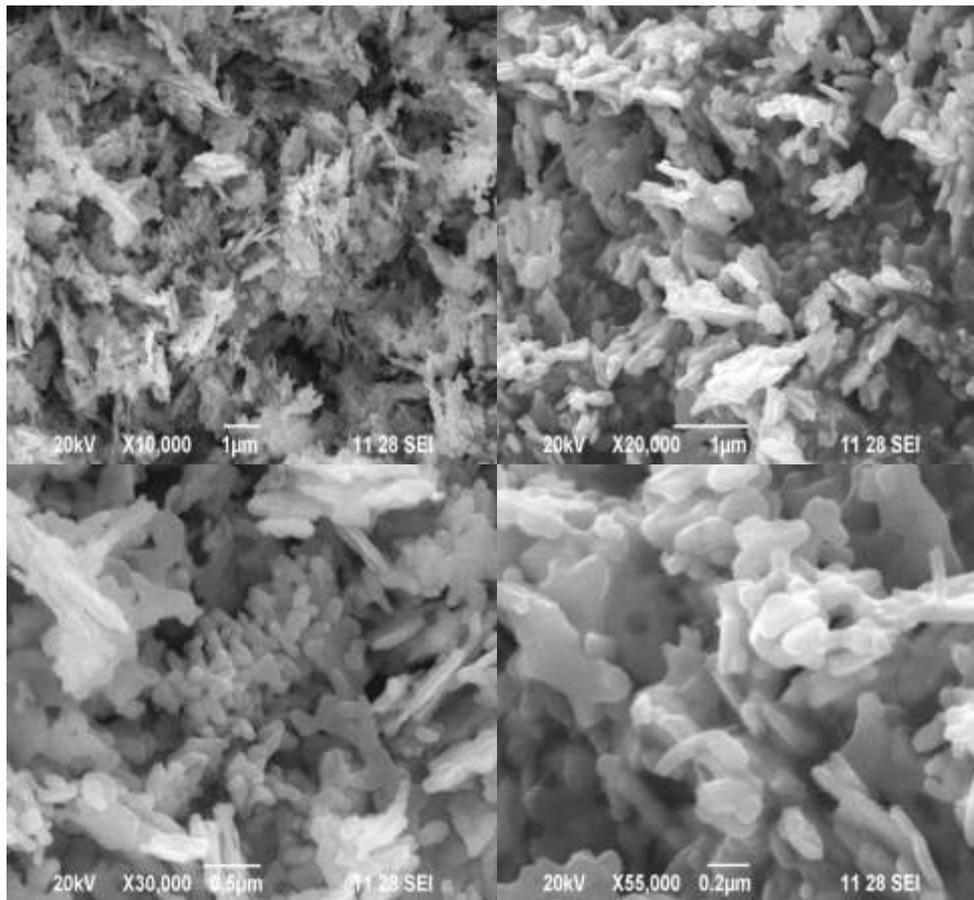


Figure 2a. Nirmala et al.

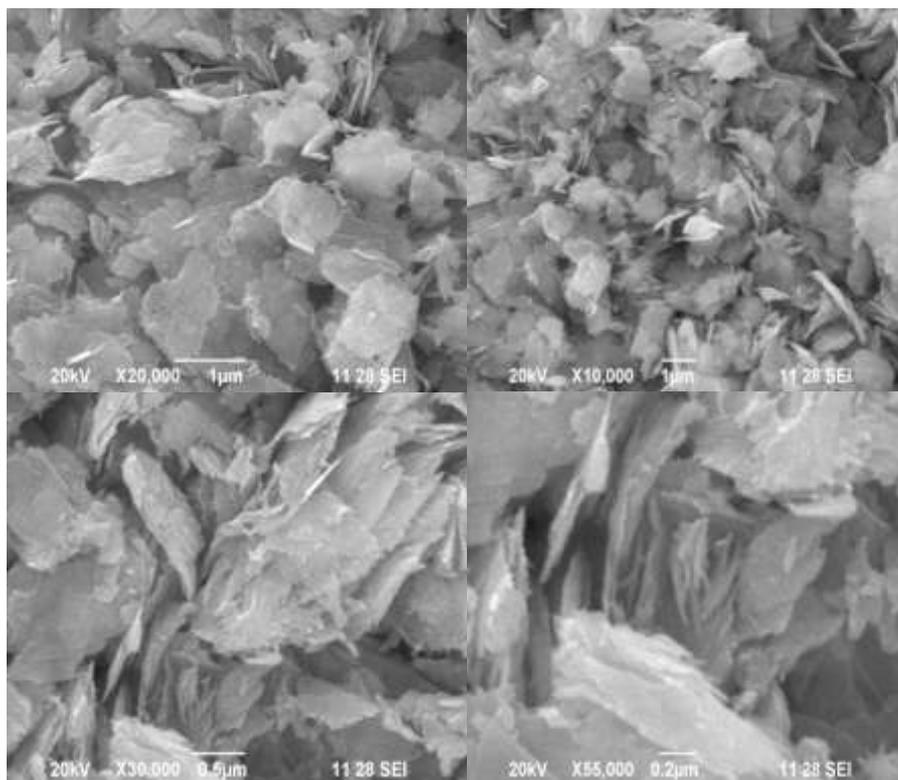


Figure 2b. Nirmala et al.

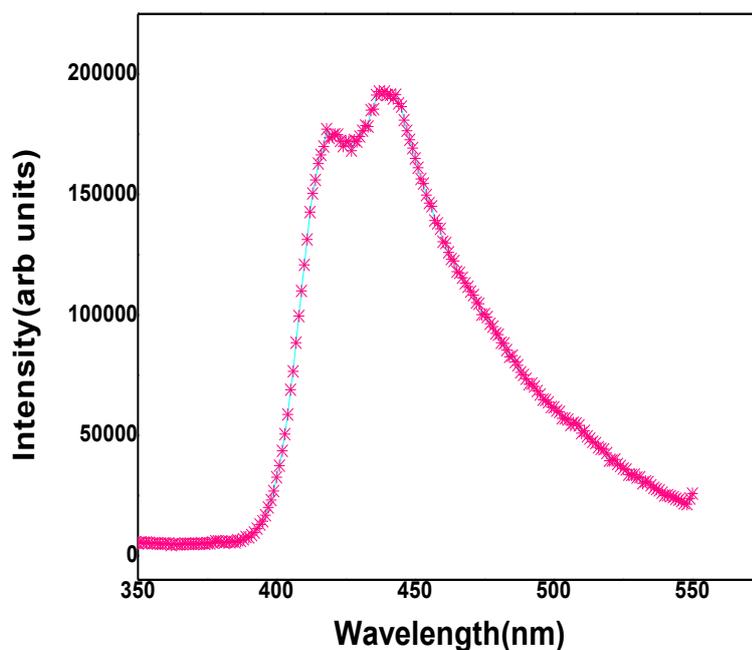


Figure 3. Nirmala et al.

Table.1 XRD Parameters of Copper Oxide Nanoparticles

Sample	JCPDS Lattice Parameters(A ⁰)			Observed Lattice Parameters (A ⁰)			Grain size (nm)
	a	b	c	a	b	c	
XRD pattern of Copper Oxide Nanoparticles	4.68	3.42	5.12	4.53	3.41	5.12	11.83
XRD pattern of Copper Oxide Nanoparticles Annealed at 500 ⁰ C	4.68	3.42	5.12	4.37	3.42	5.12	14.13