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Preparation and Characterization of Copper Oxide Nanoparticles

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Abstract: Copper oxide nanoparticles were prepared by modified sol-gel technique using sodium dodecyl sulphate as a surfactant. Effect of calcination temperature on particle size, band-gap, crystallinity and morphology of the nanoparticles were studied with the help of particle size analysis, UV-Spectroscopy, powder X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) studies. Prepared nanoparticles will be tested for their activity towards gas sensing.
Keywords*:* Copper oxide nanoparticles, modified sol-gel, surfactant, gas-sensor.

I. Introduction

Copper oxide is a semiconductor material and has a natural abundance of starting material (Cu). It is non-toxic and easily obtained by the oxidation of Cu. Copper oxide is one of the important metal oxide which has attracted recent research because of its low cost, abundant availably as well as its peculiar properties. It is used in the fields like catalysis, superconductors, ceramics as a kind of important inorganic materials etc., CuO has been used as a basic material in cuprate High-T_c superconductors as the superconductivity in these classes of systems is associated with Cu-O bondings[1]. Among all metal oxide nanoparticles, copper oxide has gained the most interest because of its wide applications, such as in solar cell technology, field emission,magnetic storage media, lithium ion batteries, gas sensing, drug delivery, magnetic resonance imaging, and field emission devices. Varieties of physical and chemical methods have been proposed to synthesize CuO nanoparticles (CuO-NPs) [2]. CuO-NPs belong to monoclinic structure system with the brownish-black appearance [3]. They find their significant role in antibacterial agents to fabrics [4]. CuO NP treatment is known to induce a disruption of the blood–brain barrier in vivo in mice and rats. Under in vitro conditions, CuO NPs were also found to induce toxic effects in different types of neuronal cells such as the human SH-SY5Y neuroblastoma and H4 neuroglioma cells [5]. CuO-NPs have been prepared with different sizes and shapes *via* several methods such as sonochemical, alcohothermal synthesis, direct thermal decomposition, electrochemical methods, colloid -thermal synthesis process, and microwave irradiation [6]. Preparation of CuO NPs via solgel technique was

optimised as well as the effect of calcination temperature on the properties of copper oxide nanoparticles was also studied and reported herein.

Experimental details

All the chemicals used were purchased from Merck Specialities chemical PVT LTD and used as such without any further purification. Sodium dodecyl sulphate [$CH_3(CH_2)_{11}OSO_3Na$] was 1.441g of Sodium dodecyl sulphate in water is allowed to stir for 10 min at room temperature. Aqueous solution of copper sulphate pentahydrate [$CuSO_4.5H_2O$] is added drop-wise into Sodium dodecyl sulphate under constant stirring. Hexamine [$(CH_2)_6N_4$] solution in water is also added to the above mixture in drop wise and pH of the solution was adjusted to 9 by the addition of aqueous ammonia [NH_3]. This solution was maintained at 160°C in an autoclave for 3h. Then, the resulted suspension was centrifuged, washed, dried at room temperature and calcinated at three different temperatures (400°C, 500°C and 600°C) and the samples were named as A,B & C respectively.

Above prepared Copper Oxide nanoparticles were characterized by the following techniques: Diffused reflectance (DRS) spectrum was recorded at room temperature with Analytic Jena spectrophotometer. The powder X- ray diffraction (XRD) patterns were recorded using PANalytical X – Ray diffractometer (Cu- Kα radiation, λ= 1.54Å) with a scanning rate of 0.02˚/sec in 2θ range from 10- 90°. The Scanning Electron Microscopic (SEM) images of the samples were recorded on JEOL JSM- 6490L A scanning electron microscope. Particle size of the materials was determined from the HORIBA particle size analyzer.

II. Results & Discussions

Particle size (Table-1) of the CuO nanoparticles (A, B & C) was found to increase with calcination temperature. This may be attributed to the agglomeration of the particles at high temperature.

Table - 1: Particle size of the Copper Oxide nanoparticles

S.No	Sample Name	Particle Size(nm)	Crystallite Size (nm)
1	A	23	21
2	B	25	22
3	C	28	24

Reflectance edge was appeared above 830 nm for all the three samples and corresponding band gap was calculated using the realation E = hc/λ and it was found to be 4.16 eV, 4.17 eV, 4.16eV respectively for samples A, B and C. From the results, a red shift is observed with calcinations temperature due to aggregation of the particles which in turn in agreement with the results obtained from DLS.

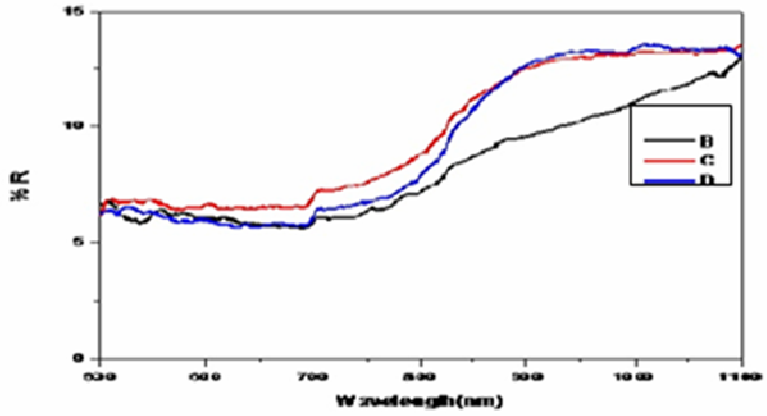

Figure-1: Reflectance spectrum of CuO NPs.

Crystalline nature of the prepared CuO nanoparticles were identified from their corresponding powder XRD patterns (figure 2). All the diffractions were well matched with monoclinic phase of CuO (standard JCPDS File No: 048-1548). Diffraction peaks with 2Θ 33.6°, 35.45°, 38.73°, 48.92°, 61.99° and 66.49° respectively were

indexed to (110), (002), (111), (020), (022) and (113) planes. Average crystallite size of the nanoparticles was calculated using Scherer equation and presented in table 1. From the results, it is inferred that the crystallite size increases with calcination temperature as evidenced from the previous results.

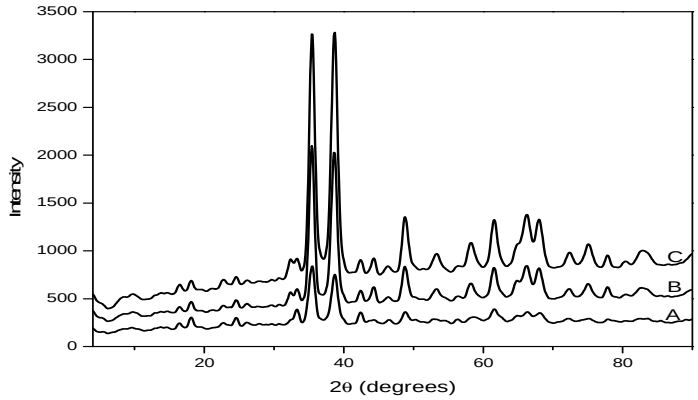

Figure -2: Powder XRD pattern of Copper Oxide *nanoparticles*

SEM image of the samples A, B & C are given in figure 3. Morphology of all of them are appeared to be porous balls and the balls were aggregated with calcination temperature. Hence the observed size increase in DLS as well as increase in crystallite size (XRD data).

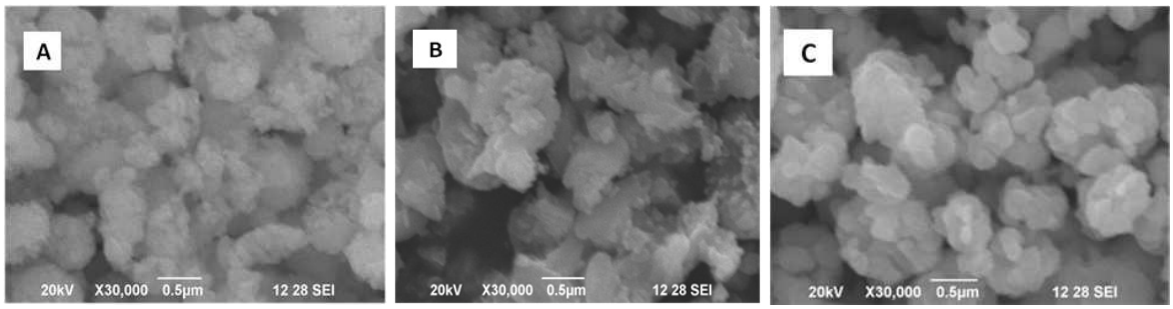

Figure - 3: SEM images of CuO NPs

Conclusions:

Copper oxide nanoparticles were synthesized using modified sol-gel method and its particle size, Band gap, crystallite size and morphology were studied with various characterization techniques. It can be noted that the size of synthesized Copper Oxide nanoparticles increase with calcination temperature due to agglomeration at elevated temperatures and there is slight shift in the bandgap of the material. These prepared nanoparticles will be tested for their activity towards the sensing of oxidizing and reducing gases in future.

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