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Copper Oxide Nanoparticles Prepared by Solid State Thermal Decomposition: Synthesis and Characterization

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ABSTRACT

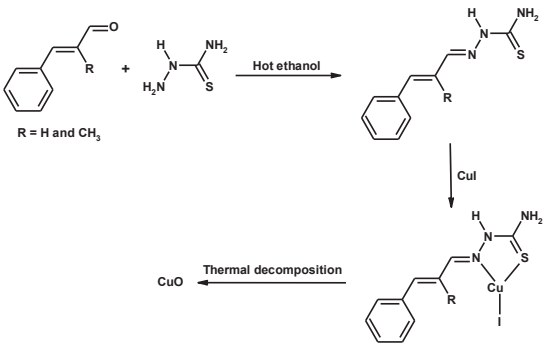
In this paper, we have focused on the preparation and characterization of copper oxide nanoparticles by solid state thermal decomposition of copper(I) iodide in the presence of thiosemicarbazone ligands without the need for a catalyst, employing toxic solvent, template or surfactant and complicated equipment, which makes it efficient, one-step, simple and environment-friendly. CuO nanoparticles were achieved at 600 ˚C for 3 h as black products and characterized by Fourier transform infrared spectroscopy (FT-IR), X-ray powder diffraction (XRD) and transmission electron microscopy (TEM). The FT-IR spectra of black powders prepared show absorption maxima at ≈ 525 cm^{-1} which are due to Cu-O stretching mode. Also, all the X-ray diffraction peaks could be readily assigned to those of crystalline CuO. The absence of any residual ligand traces or other phases in the FT-IR spectra and XRD patterns confirmed the preparation of high purity and single phase copper oxide nanoparticles. The TEM images show that the synthesized copper oxide nanoparticles are of plate like shape with average diameters of 10 – 20 nm. On the basis of the above results, the use of thiosemicarbazone ligands at the presence of suitable transition metal ions is potentially capable of forming other transition metal oxide nanoparticles by solid state thermal decomposition.

Keywords: *Copper(I) Iodide; CuO nanoparticles; Thiosemicarbazone.*

1. Introduction

Copper oxide, a p-type semiconductor with a band gap of 1.2 eV, has a great significant properties and applications such as optical properties [1,2], gas sensing [3], antioxidant and antibacterial [4], photo-electrochemical water splitting [5], electrochemical determination of dopamine [6], electro-catalytic [7], dissolution of methane in water [8] and application in lithium ion batteries [9]. Widespread applications of CuO nanoparticles insisted several methods of preparation and characterization of CuO nanoparticles viz. electrochemical-thermal [10], solvothermal [11,12] thermal decomposition [13-15], mechano-chemical oxidation [16], microwave-assisted [17] and precipitation techniques [18-20]. Among the various methods available for controlled synthesis of CuO nanoparticles, thermal methods are effective with well-controlled shapes and sizes [10-15]. Also, the other techniques are not simple, more costly and involve more reagents.

Herein, we report the synthesis and characterization of CuO nanoparticles by solid state thermal decomposition as a simple, cost effective and eco-friendly method, of copper(I) iodide in the presence of thiosemicarbazone ligands at 600˚C for 3 h (Scheme 1).



Scheme. 1- Schematic illustration of the formation of CuO nanoparticles.

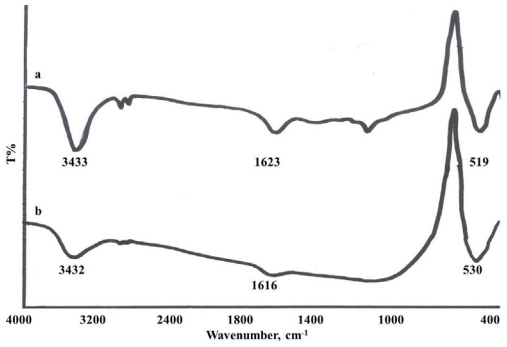


Fig. 1- The FT-IR spectra of CuO nanoparticles prepared from copper(I) iodide in the presence of catsc (a) and mecatsc (b).

2. Experimental

2.1. Materials and characterization

All reagents and solvents employed were commercially available and used as supplied without further purifications. Thiosemicarbazone ligands, catsc = 3-phenylpropenalthiosemicarbazone and mecatsc = (1E,2E)-2-Methyl-3-phenylacrylaldehyde thiosemicarbazone, are prepared following literatureprocedure [21,22]. FT-IR spectra were recorded from KBr disk on a Perkin–Elmer. X-ray powder diffraction (XRD) pattern of the CuO nanoparticles are recorded on a Bruker AXS diffractometer D8 ADVANCE with Cu-Kα radiation with nickel beta filter in the range $2\theta = 10^{o}$–80^{o}. The transmission electron microscopy (TEM) images were obtained from a JEOL JEM 1400 transmission electron microscope with an accelerating voltage of 120 kV.

2.2. Preparation of CuO nanoparticles

CuI (1 mmol) and thiosemicarbazone ligands (1 mmol) were dissolved separately in 10 mL acetonitrile. Then, mixed and stirring for 30 min. The yellow products filtered, washed with acetonitrile and dried at 70 °C in an oven. Then, the yellow products are taken in a crucible and placed in a muffle furance and heated to 600°C at a rate of 10°C/min in air. Nanoparticles of CuO are produced after 3 h, washed with ethanol and dried at room temperature. The synthesized CuO nanoparticles are characterized by FT-IR, XRD and TEM.

3. Reults and discussion

The FT-IR spectra of the synthesized CuO nanoparticles following solid-state thermal decomposition are presented in Fig. 1. Broad peaks at approximately 3421 and 1610 cm^{-1} are attributed to H-OH stretching [6]. The vibration frequencies at 519 cm^{-1} and 530 cm^{-1} in the FTIR spectra of CuO nanoparticles have been assigned to Cu-O stretchings [6]. Existence of starting reactants or impurities is ruled out by the absence of stretching vibrations for CH, C=N and C=C functionalities.

The structure and the phase composition of CuO nanoparticles, obtained after calcination at 600 °C for 3 h, have been ascertained by powder XRD analysis (Fig. 2). All diffraction peaks can be well indexed to the monoclinic structure of copper oxide (JCPDS database No. 05-0661) [6,7,9]. Absence of any impurity peaks from other phases indicates its high purity. XRD results are in close agreement with the FTIR spectra. Broadening of the diffracted lines indicates the higher crystallinity of CuO nanoparticles [9].

The detailed morphology and structures of CuO nanoparticles are further characterized by TEM (Fig. 3) images which clearly indicate similar morphologies and size of CuO nanoparticles. They also indicate that CuO nanoparticles remain agglomerated having almost uniform in size.

4. Conclusion

In summary, pure CuO nanoparticles having similar morphologies and size, have been prepared by solid-state thermal decomposition of copper(I) iodide at the presence of thiosemicarbazone ligands at 600 °C. Absence of any complex or other phases

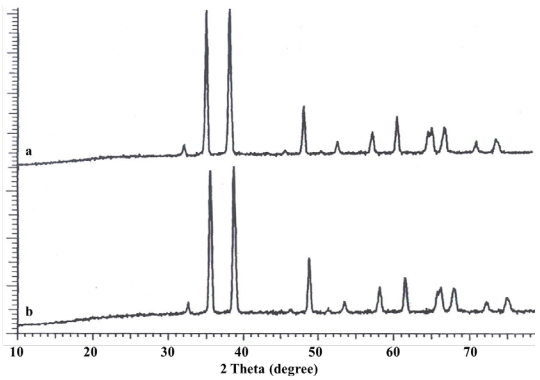


Fig. 2- XRD patterns of CuO nanoparticles prepared from copper(I) iodide in the presence of catsc (a) and mecatsc (b).

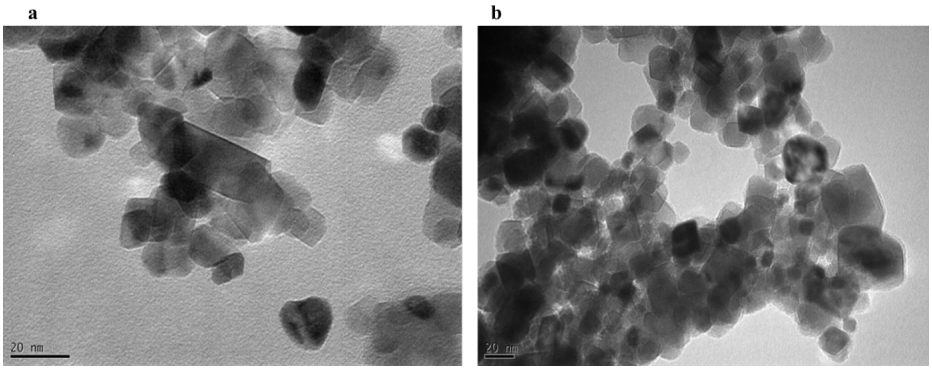


Fig. 3- TEM images of CuO nanoparticles prepared from copper(I) iodide in the presence of catsc (a) and mecatsc (b).

of copper oxide nanoparticles assures their high level purity. This method is highly reproducible and may be useful for preparation of other transition metal oxide nanoparticles.

Acknowledgements

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