Characterization of Copper Oxide Nanocrystal by TEM, XRD and FTIR

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Abstract: Copper oxide nanocrystal has been prepared and characterized through studies of TEM, FTIR and XRD. The TEM images taken for different nanocrystal reveal that the size of synthesized sample is in the range 5nm to 15nm and the size of prepared CuO nanorod varies from 6 micrometer to 15 micrometer in length and 10 nanometer to 30 nanometer in width FTIR studies show that synthesized nanocrystals comprises purely CuO without any trace of Cu_2O. The XRD data demonstrates the growth of nanocrystal with increase of thermal treatment temperature.
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Keywords: Nanocrystal; X-ray diffraction ;Fourier transform infrared spectroscopy; Transmission electron microscope.

1.Introduction

Transition metal oxides have attracted much attention in recent years because of their size dependent optical properties and electronic structure [1, 2]. Copper oxide is a transition-metal oxide with a monoclinic structure [3, 4]. It is a covalent semiconductor having an indirect band gap between 1.2eV and 1.5 eV. Controlling the size, shape and structure of nanocrystals is technologically important because it will have strong effect on the optical, electrical, and catalytic properties. With the decreasing crystal size, CuO nanocrystals exhibit some unique properties like change in ionic character, ferromagnetic response etc. Investigation of electronic properties of CuO nanocrystals will provide more insight into the electronic correlation and also the electronic coherent states[5, 6, 7]. As Copper oxide has more industrial applications like solar energy storage, semiconductors and catalysis, it attracts researchers to study its behavior at various size regimes. Copper(II) oxide or Cupric oxide (CuO) is the higher oxide of copper. As a mineral, it is known as tenorite. Copper oxide belongs to the monoclinic crystal system, with a crystallographic point group of 2/m or C_{2h}. The space group of its unit cell is C2/c, and its lattice parameters are a = 4.6837(5), b = 3.4226(5), c = 5.1288(6), $\alpha = 90°$, $\beta = 99.54(1)°$, $\gamma = 90°$. The copper atom is coordinated by 4 oxygen atoms in an approximately square planar configuration as shown in the figure-1 and figure-2.

Cupric oxide is used as a pigment in ceramics to produce blue, red, and green (and sometimes gray, pink, or black) glazes. It is also used to produce cuprammonium hydroxide solutions, used to make rayon. It is also occasionally used as a dietary supplement in animals, against copper deficiency. Copper (II) oxide has application as a p-type semiconductor, because it has a narrow band gap of 1.2 eV.

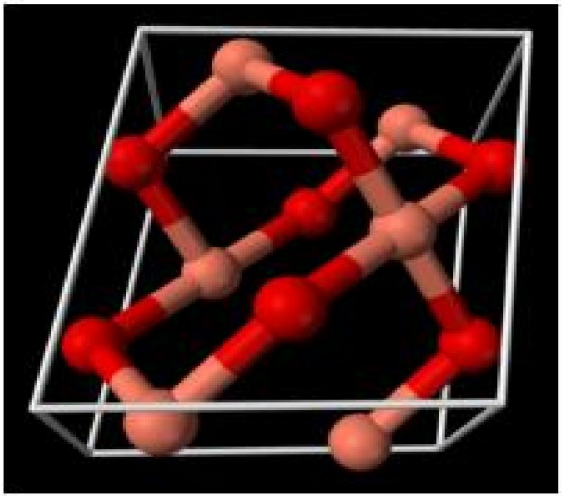

Fig-1: The unit cell of copper(II) oxide

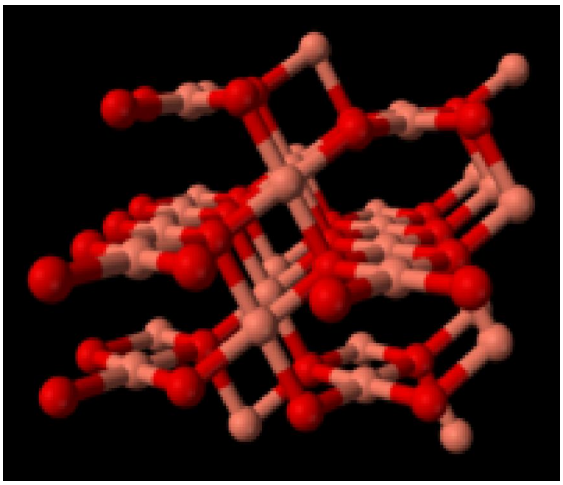

Fig-2: Part of the crystal structure of CuO

It is an abrasive used to polish optical equipment. Cupric oxide can be used to produce dry cell batteries [8, 9, 10]. It has also been used in wet cell batteries as the cathode, with lithium as an anode, and dioxalane mixed with lithium perchlorate as the electrolyte. Copper(II) oxide can be used to produce other copper salts. It is also used when welding with copper alloys. Another use for cupric oxide is as a substitute for iron oxide in thermite. This can turn the thermite from an incendiary to a low explosive.In view of its wonderful application in different fields of science and technology by TEM, XRD and FTIR.

2. Experimental work.

The experimental work has been done in Raman Research Institute, Banglore.

2.1 Preparation of Copper Oxide Nanocrystals and nanorods.

The synthesis of Copper oxide nanocrystals is done by a combined precipitation-pyrolysis method, which involves initially preparing precursors and finally decomposing the precursors in a furnace with different annealing temperatures, which lead to the final products of copper oxide nanocrystals.

2.2 Procedure for Preparing Precursor A

Preparation of precursor A involves the following reaction. 0.3 M of aqueous ammonium carbonate is prepared by dissolving 4.714 g of ammonium carbonate in 100 ml distilled water. Similarly, 0.05 M of aqueous copper acetate is prepared by dissolving 4.991g of copper acetate in 500 ml of distilled water. Now, 50 ml of freshly prepared aqueous ammonium carbonate is rapidly added to 300 ml of aqueous copper acetate, and precipitate is formed. After a reaction time of 1minute, the precipitate formed is separated by a centrifuge process. Then they are washed with distilled water and ethanol to remove possible remnant ions in the final products, which are dried in air at 60^0 C and kept ready for further reaction.

2.3 Procedure for Preparing Precursor B

0.3 M of aqueous sodium hydroxide is prepared by dissolving 4.714 g of sodium hydroxide in 100 ml distilled water. Similarly, 0.05M of aqueous copper acetate is prepared by dissolving 4.991 g of copper acetate in 500 ml distilled water. 50 ml of prepared aqueous sodium hydroxide is mixed rapidly with 300 ml of aqueous copper acetate. After a reaction of 1 min the precipitate is formed which is separated by centrifuge process, and then washed with distilled water and ethanol. It is then dried in air at 60^0 C.

2.4 Thermal Decomposition of Precursors

Thermal decomposition of the precursors in a furnace with different annealing temperatures led to the final product of CuO nanocrystals and nanorods. Sample (S2) was prepared at 200^0C by using precursor A under constant nitrogen flow. Annealing at temperatures 300^0C, 400^0C, and 500^0C, samples (S3), (S4), and (S5) are prepared respectively from precursor A. without nitrogen flow the final product obtained was copper oxide nanocrystals. Sample (S6) was prepared by using precursor B with the reactant copper sulphate at an increased concentration 0.15 M, which is obtained by dissolving 3.742g of copper sulphate in 100 ml of distilled water and at an annealing temperature of 300^0C. Here the final product obtained was copper oxide nanorods.

3 Results and Discussion

3.1 Transmission Electron microscope

The TEM image is taken for different CuO nanocrystals samples prepared at different annealing temperaures which reveals the fact that the size of the synthesized CuO nanocrystals where in the range between 5nm to 15nm.

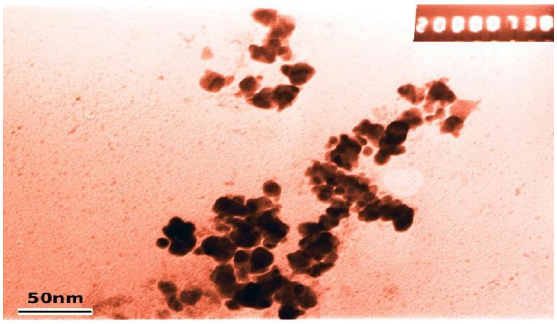

Figure 3 :TEM image of CuO nanocrystals.

Also, the TEM image showed that the size of the prepared CuO nanorod were of 6 to 15 micrometer in length and 10 to 30nm in width. The TEM images of the CuO nanocrystals is shown in the figure -3 and figure -4 and CuO nanorod in Figure-5.

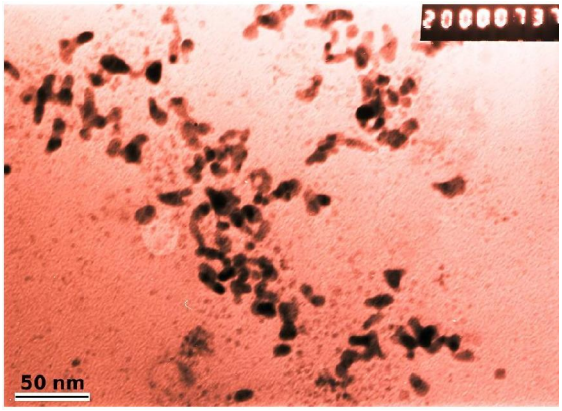

Figure 4 :TEM image of CuO nanocrystals.

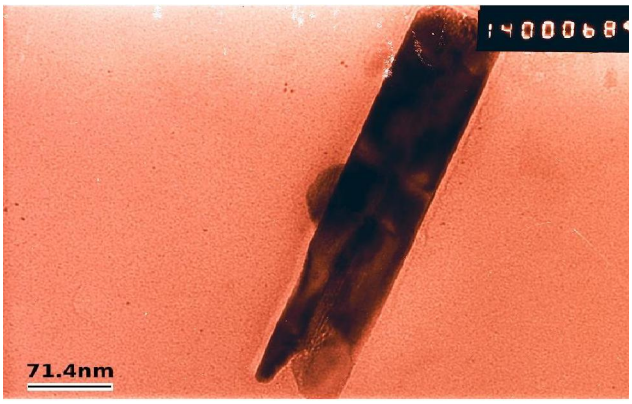

Figure 5: TEM image of CuO nanorods.

3.2 Fourier Transmission Infrared Spectroscopy Studies

The FTIR transmission spectra of CuO nanocrystals were taken at room temperature. The three vibrational modes are observed at 573 cm^{-1}, 523 cm^{-1} and 452 cm^{-1} these broad peaks are particular for CuO –II nanocrystals. Which is shown in Figure-6 and figure-7 The high frequency mode at about 573 cm^{-1} is reported to be due to Cu – O stretching. Moreover, modes due to Cu_2O are not seen, as infrared active modes of Cu_2O appear at 610 cm^{-1} This reveals that the synthesized nanocrystals comprises purely CuO phase without any trace of Cu_2O.

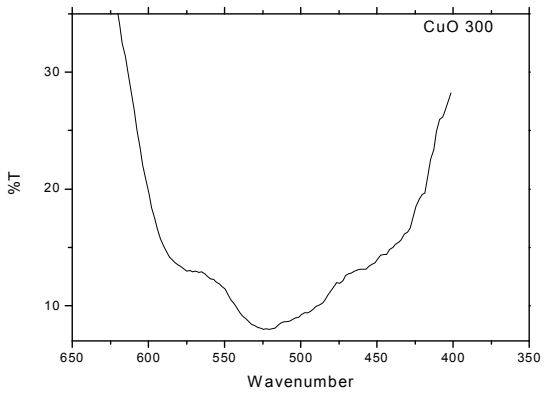

Figure 6: FTIR picture of CuO nanocrystal

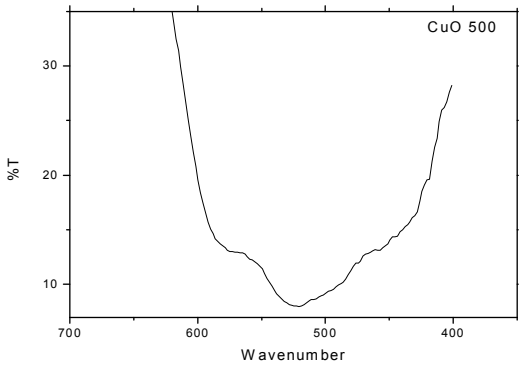

Figure 7: FTIR picture of CuO nanocrystal

3.3 X-Ray Diffraction Studies

The mean crystalline sizes of the samples estimated using scherrer formula and are in the range of 5 to 15nm. The XRD data clearly demonstrate the growth of nanocrystals as the thermal treatment temperature increases. The FWHM increases as the sample size decreases. The XRD results of the prepared samples are shown in figure-8 and figure-9.

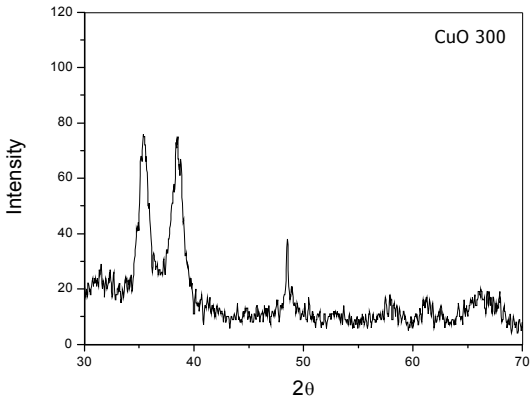

Figure 8: XRD pattern of sample prepared at 300 ^{0}C

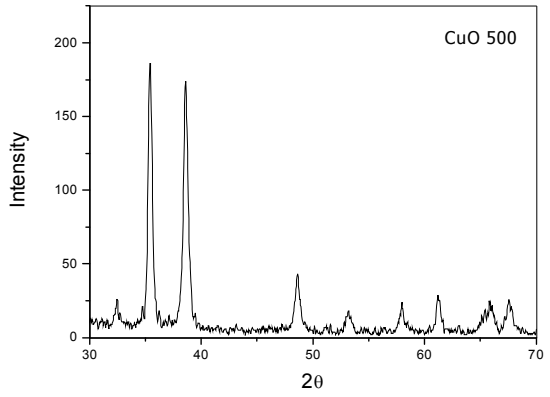

Figure 9: XRD pattern of sample prepared at 500 ^{0}C

4. Conclusion

Thus from TEM study, we conclude that size of synthesized CuO nanocrystal is in the range 5nm to 15 nm. FTIR studies reveal that synthesized nanocrystal comprises purely CuO without any trace of Cu_2O. XRD data demonstrates the growth of nano crystal with increase of thermal treatment temperature.

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