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Preparation of Nanostructured Copper Oxide Rods Using Advanced Sonication Method

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Abstract

In recent times, copper-based nanomaterials have received lots of attention because of their potential applications as gas sensors, magnetic storage media, solar energy transformation, semiconductors and organic catalyst. The aim of present work is to synthesize CuO nanorods by using inventive sonication method and to characterize by powder XRD, particle size analyzer and TEM. The powder XRD was used for the structural identification and unit cell parameter determination of nanorods. Formation of single phasic CuO nanorods was confirmed from XRD. TEM and particle analyzer verified the nano size and shape of CuO. Nanorods were formed by preparation of CuO using sonication which was confirmed from the TEM analysis.

Keywords

Sonication, CuO Nanorod, TEM

1. Introduction

In modern materials science and technology, synthesis and properties of one-dimensional nanostructured materials is one of the most important areas. Nanoscale materials are not only employed to study physical properties but can also be utilized for fabrication of electronic and optical nano-devices. Metal oxides are of great interest in research due to various properties from wide band-gap insulators to metallic and superconducting which creates a significant interest in the synthesis of metal oxide nanomaterials. Demanding research efforts have been devoted to the synthesis and investigation of the physical and chemical properties of semiconducting oxides such as CuO, ZnO, SnO_2 and TiO_2. Among semiconducting oxides, cupric oxide (CuO) is the important semiconducting oxide with a monoclinic structure and direct band gap of 1.85 eV [1]. CuO has potential applications in sensors [2], catalysis [3], solar cells [4], electrode materials [5], magnetic storage media [6], lithium ion batteries [7] and high T_c superconductors [8]. As compared to other transition metal oxides like ZnO, SnO_2, TiO_2 and Fe_2O_3, synthesis and characterization of CuO nanoparticles is the least explored area which makes it an interesting candidate to investigate its properties. Moreover, the doping of the semiconductor nanoparticles with impurity metal ions is one of the most important methods to modify the characteristics of the material. Using various dopants the improvement in material's characteristic properties have been reported by many authors. Ahmed et al. observed the band gap narrowing of Ni doped SnO_2 nanoparticles [9], The tuning of emission properties of Mn doped Cu_2O nanoparticles reported by Das et al. [10]. Till now the study of Mn doped CuO is a least explored area.

2. Experimental Method

Solution A of 0.38M copper acetate [$Cu(CH_2COO).2H_2O$-molecular weight 199 g/mol] was prepared by dissolving 7.586g $Cu(CH_2COO).\ 2H_2O$ in 100ml ethanol [CH_3CH_2OH] with stirring for 1 hour. Solution B of 0.25M sodium hydroxide [NaOH - molecular weight 40 g/mol] was prepared by dissolving 10g NaOH in 100ml distilled water with stirring for 1 hour. After sonification for 1 hour, drop wise add solution B to solution A until the solution becomes bluish color. Afterward particles were washed for two times with CH_3CH_2OH (ethanol) using centrifugation then further washed with double distilled water. Finally, brown colored nanoparticles were obtained. But if we want growth of nanorod, we need to change the washing process. Process like, we washed it with ethanol and stayed for 5 days and particles or rod settled. Furthermore, repeated this process for two times. And finally we washed with distilled water and dry it in microwave at 80°C for 5 hours. We got nanorod like particles which is confirmed in TEM images.

3. Results and Discussions

Here we found the single crystal nanostructure of CuO, characterized by x-ray diffraction (XRD) at room temperature using Cu - K_α radiation. From figure 2, it is clear that material exhibit monoclinic structure. To satisfy the monoclinic structure, here we had calculated lattice parameters: a = 4.6837 Å, b = 3.4226 Å, c = 5.1288 Å and also α = 90°, β = 99.54°, γ = 90°, which was matched with the reported data [11] and (JCPDC No. 96-410-5686). In this chapter, we also calculated the particle size with the help of Scherrer's formula [12]:

$$P = \frac{0.9\,\lambda}{\beta\ cos\theta}$$

where, P = crystallite size, λ = wavelength (1.54Å), β = full width half maxima, θ = diffraction angle. It had been evaluated by taking instrumental as well micro strain broadening which has crystallite size about 17.5nm.

From the particle size analyzer which works on the basis of laser diffraction technique, we observed that the particles size varies from 14 nm to 44 nm, which is shown in figure 3. In plot, x axis show particle size whereas y-axis show number of particles. Suspended solution was made in double distilled water and alcohol solutions, which settled after four days due to that nearly equal particle size was obtained.

The size and morphology of the CuO nanoparticles were analyzed by transmission electron microscopy (TEM). The TEM image (fig. 4) reveals that the product consists of rod like particles with a regular morphology. The size of the particles observed in the TEM image is in the range of 13-18nm. Increase in NaOH ratio gave sheet like particles which is in good agreement with estimation from Debye-Scherrer [13]. By increasing or decreasing the temperature of nanostructured CuO, rod like particle structure can be modified [14]. In fig. 4 (a) & (f), observed lattice fringes used to measure the distance between two planes.

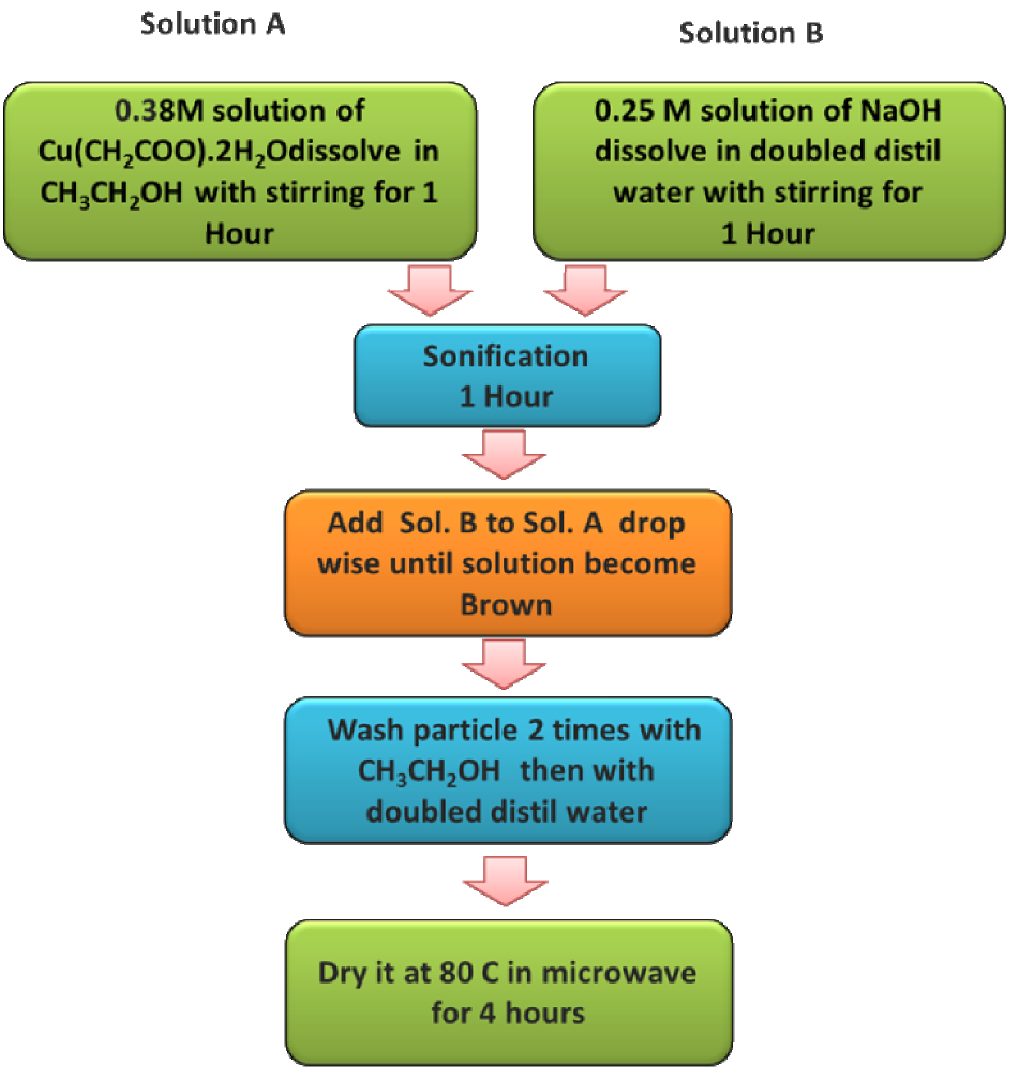

Figure 1. *Flow chart for preparation of CuO nanorods.*

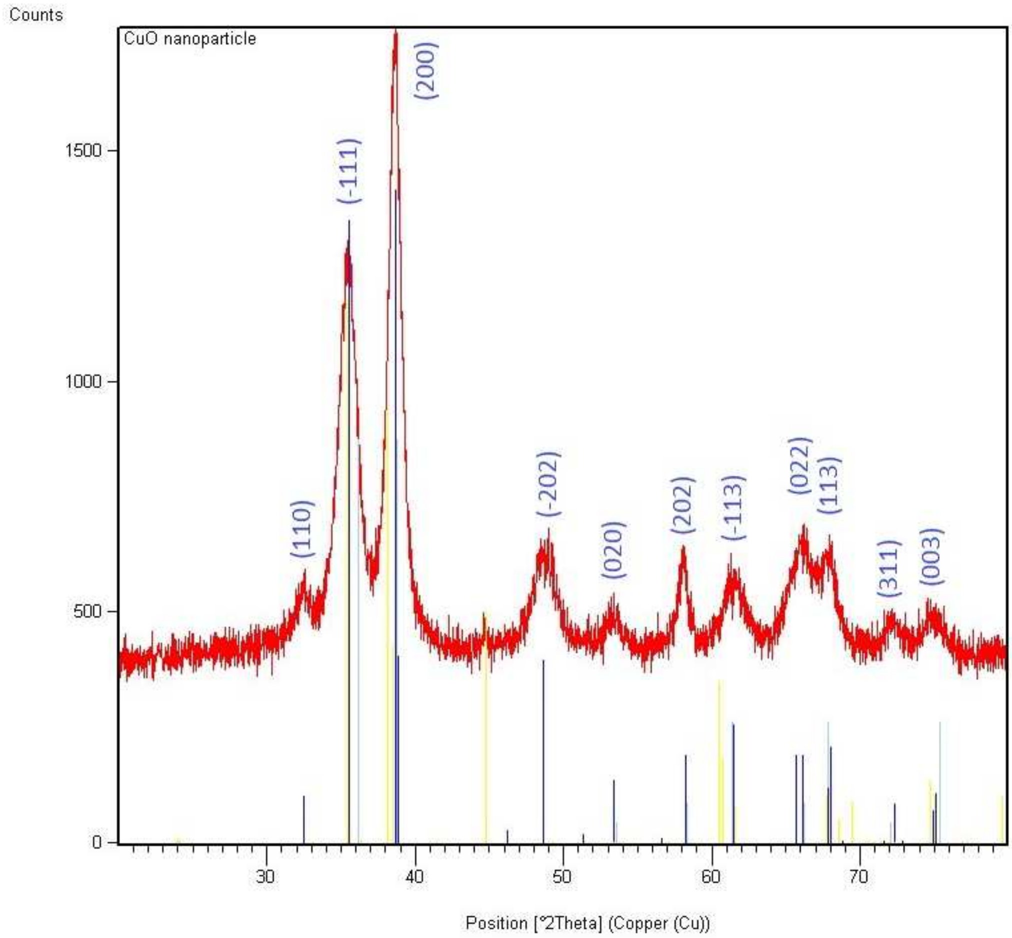

Figure 2. *XRD pattern of copper oxide nanorods.*

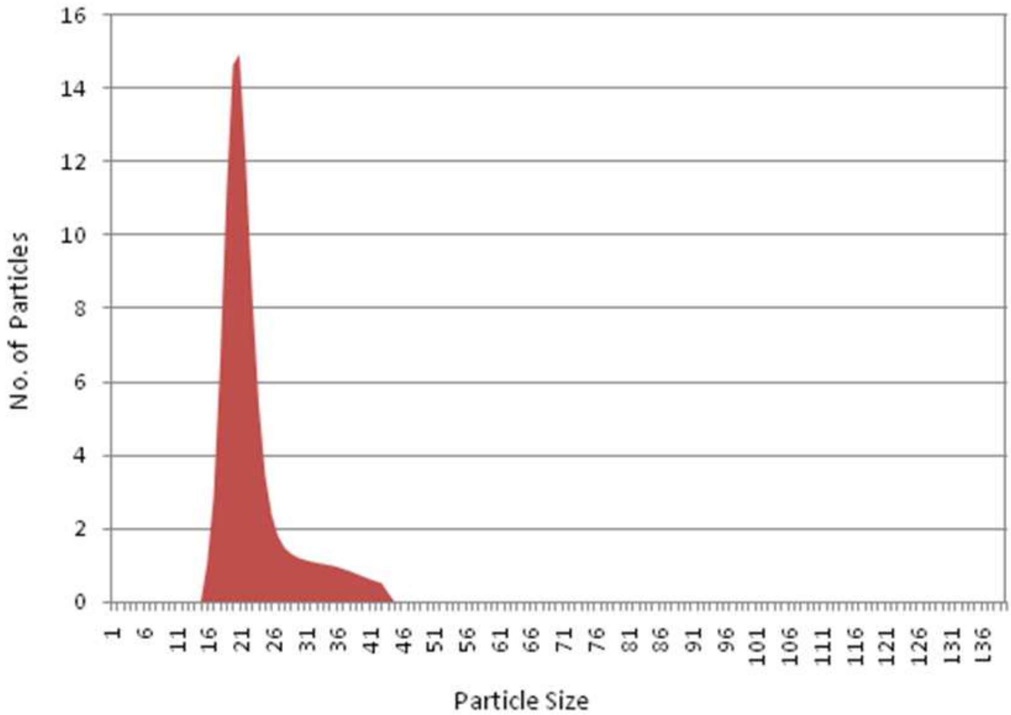

Figure 3. *Particle size analysis of CuO nanorods.*

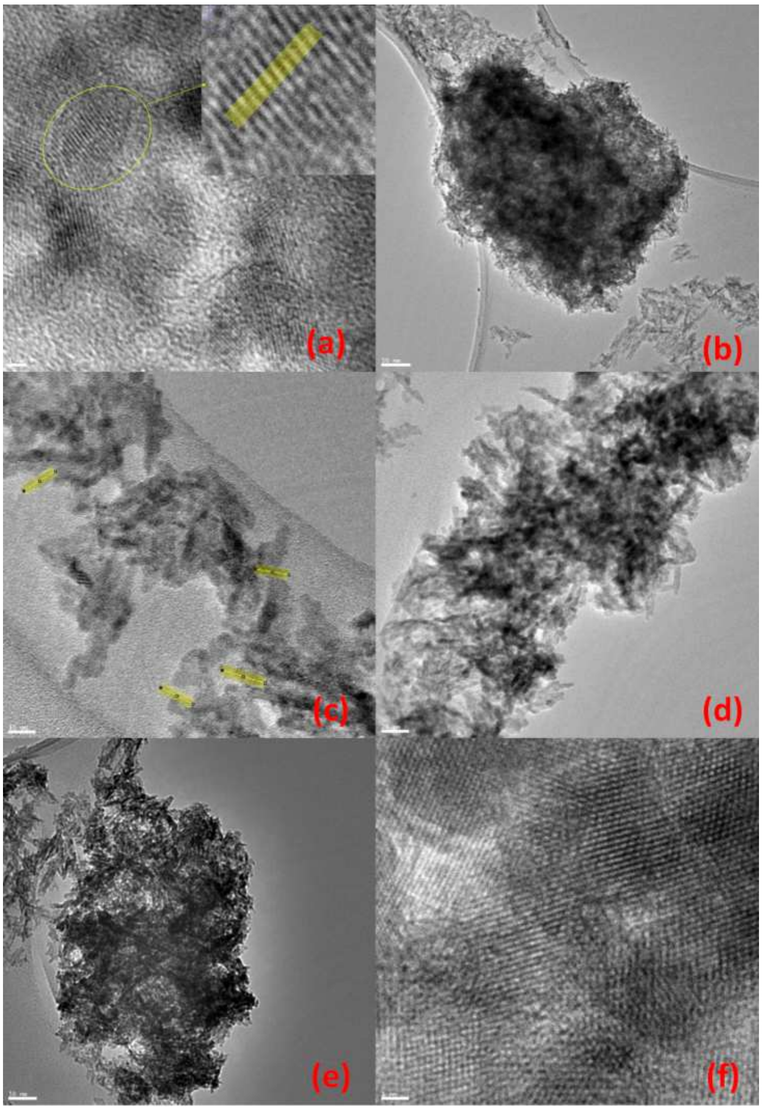

Figure 4. *TEM image of nanorods of CuO.*

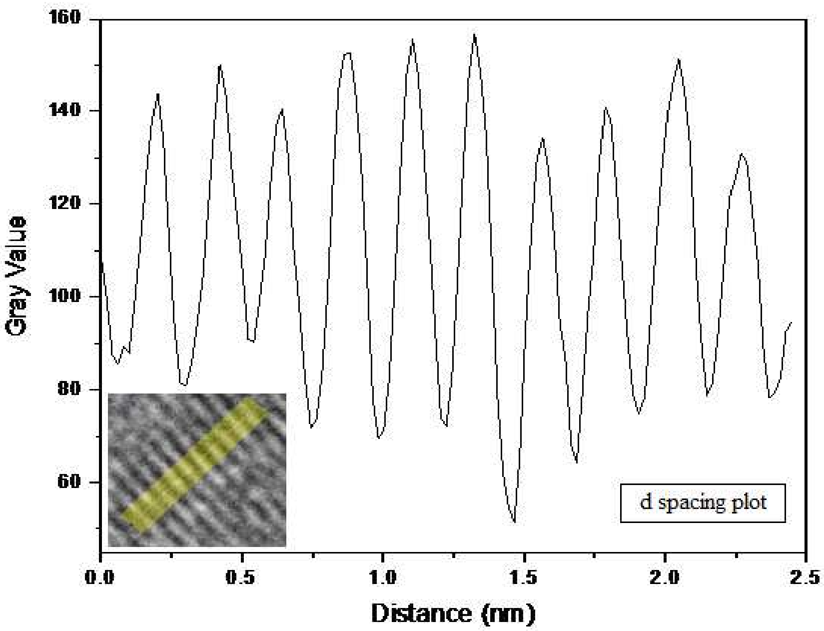

Figure 5. *D spacing analysis of CuO nanorods from TEM image.*

From the above plot (fig. 5), d spacing of material can be measured. The plot to determine d spacing was drawn with the help of the software named image j. Spacing between two peaks shows the d spacing value of the material and was found by averaging which has d spacing value ~0.230015 nm (2.30015Å).

4. Conclusions

It can be concluded that CuO nanorods can be synthesized successfully using sonication method which is proved to be effective technique to prepare nanorods. Monoclinic phase without any impurity phase of CuO nanorods was affirmed from XRD. Particle size was evaluated by using two different measurement tools. Firstly it was measured by particle size analyzer which gave particles size around 14 to 44nm. Then measurement was done by TEM which gave clear vision of particle size and also show formation of nanorods of CuO.

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