

Synthesis of CuO Nanoparticles by using Simple Precipitation Method

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Abstract: *Copper Oxide nanoparticles were synthesized by simple precipitation method using copper sulphate and sodium hydroxide as a cationic and anionic precursors. Structural and optical properties were investigated by using XRD, SEM and Uv-Visible spectroscopies. The XRD studies revealed that the as-synthesized CuO nanoparticles were monoclinic structure along with the (002) plane preferential orientation with fine crystalline in nature. HRSEM studies discovered the surface morphology of the CuO nanoparticles. As compared to the other nanoparticles, CuO that was synthesized through simple precipitation method demonstrated some amplification in the photocurrent. Accordingly it demonstrates that the CuO nanoparticles could be one of the hopeful candidates for the photoelectrochemical performance.*

Keywords: Copper Oxide, Nanoparticle, Precipitation method, Photocurrent

1. Introduction

Among various p-type semiconductors copper oxide is one of the potential semiconductor. Due to its tremendous optical, electrical and magnetic properties it gains considerable attention. The tapered band gap of CuO has 1.2 eV which is extensively used in diverse applications such as catalysis [1], solar energy conversion [2], gas sensor [3] and field emission [4]. However, these novel properties can be improved by synthesis in CuO nanostructures that shown excellent performance comparing to bulk counterpart. Different nanostructures of CuO are synthesized in form of nanowire, nanorod, nanoneedle, nano-flower and nanoparticle. In the past decades, several methods have been situated to prepare CuO nanoparticles with different sizes and shapes such as thermal oxidation [5], sonochemical [6], combustion [7] and quick-precipitation [8-9].

Among these techniques, precipitation method is a facile approach, which attracts considerable attention in industries because of low energy and temperature, inexpensive and cost-effective approach for large scale production and good yield.

In the present work, the main objective is to investigate the structural properties of CuO nanostructures synthesized via precipitation method and annealing process. Copper nitrate and NaOH were taken as a cationic and anionic precursor. The as-prepared CuO were analyzed by scanning electron microscopy, X-ray diffractometer.

2. Experimental Section

2.1 Chemicals

Copper sulphate heptahydrate ($\text{CuSO}_4 \cdot 7\text{H}_2\text{O}$) and Sodium Hydroxide (NaOH, 99%), were procured from sigma-Aldrich, India. All of these chemicals were AR grade and used directly without any further purification.

2.2 Synthesis of CuO Nanoparticles

The Copper Oxide nanoparticles were synthesized from an aqueous medium by a simple precipitation process. Initially, the reaction bath contained the cationic precursor solution of $\text{CuSO}_4 \cdot 7\text{H}_2\text{O}$, which was dissolved by using double distilled water and the precursor solution was magnetically stirred for several minutes at constant bath temperature of 60°C. With that, the solution of anionic precursor (NaOH) is added in a drop wise manner. Black precipitates were obtained and repeatedly washed by using deionized water. Subsequently, the washed precipitates were dried at 80 °C for 12 hours. Finally, the precipitates were calcined at 400 °C and used for further characterization.

2.3 Characterizations

The structural property of the nanoparticles were studied by XRD with a (XPRT-PRO), in the range of 2θ were 20 to 80° with Cu K_α ($\lambda = 1.5406$ nm) radiation. The surface morphology was investigated by high-resolution scanning electron microscopy (HRSEM) using quantum 200 FEGE model, operating at an accelerating voltage of 10 kV. A transient photocurrent study was tested through a 100 W Xenon lamp (OSRAM, Germany) used as light source.

3. Results and Discussion

3.1 XRD Studies

The crystallinity and crystal structure of the as synthesized CuO nanoparticles were studied by XRD measurement. Fig.1 shows the XRD patterns of the synthesized CuO nanoparticles. From the results the presence of enhanced diffraction peaks for CuO corresponding to (002) (200) and (202) planes indicated the configuration of pure monoclinic phase are well reliable with the JCPDS no. 80-1268. Also, no other diffraction peaks are obtained for impurity and other compound, in this diffraction peaks proved that CuO nanoparticles are well crystalline nature. Good crystallinity is received by using CuSO_4 as a cationic precursor

compared with other cationic sources of copper.[10]. From the enhanced (hkl) planes, the lattices constant (a) were evaluated using the following equations,

$$a = d (h^2 + k^2 + l^2)^{1/2} \quad (1)$$

where d is inter-planar spacing of the atomic planes. The calculated d-spacing values are used for determination of lattice constant. The average crystallite sizes have been calculated by Scherrer formula with using the full width at half maximum (FWHM) value of preferentially oriented diffraction peaks.

$$D = 0.9 \lambda / (\beta \cos\theta) \quad (2)$$

where β is the FWHM of the diffraction line in radians and λ is the X-ray wavelength. The calculated average crystallite sizes were originate in the range between 10-40 nm . The micro strain (ϵ), dislocation density (δ) and have been resolved through the following relations (Equations 3 and 4).

$$\text{Micro strain } (\epsilon) = \beta \cos\theta / 4 \quad (3)$$

$$\text{Dislocation density } (\delta) = 15\epsilon / aD \quad (4)$$

where θ is the Bragg's angle and δ is common physical phenomena of thin films and ϵ is the strain which induces a deformation in one part per million.

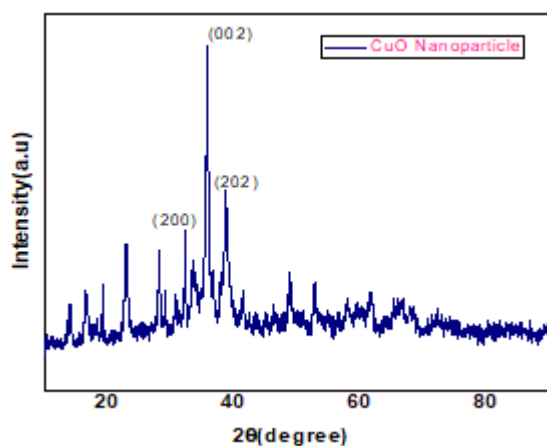


Figure 1: XRD spectra of as-synthesized CuO Nanoparticles

3.2 Scanning Electron Microscopy

In order to examine the surface morphology of CuO nanoparticles, the as-synthesized nanoparticles were subjected to High Resolution Scanning Electron Microscopic studies. Figure 2 displayed SEM image which illustrating the morphology of CuO-nanoparticles prepared via the precipitation method. As-prepared CuO precipitation using copper sulphate and sodium hydroxide are in good dispersion and found to be petite nanorod-like structure and some of nanoparticles were agglomerated which may due to different sizes of nanoparticles present in it.

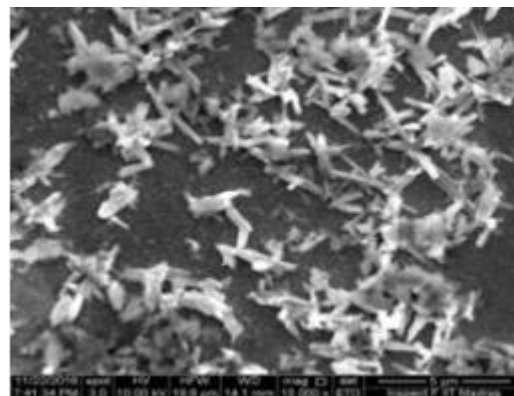


Figure 2: HRSEM image of as-synthesized CuO Nanoparticles

3.3 Photocurrent studies

The transient photocurrent study was carried out to evaluate the photostability of the CuO nanoparticles. To attain a deeper insight into the behaviour of photogenerated charge carriers, the profiles of CuO nanoparticles were obtained from the Copper sulphate and NaOH as the cationic and anionic precursors of as-synthesized CuO. Figures 3 shows photocurrent response of CuO nanoparticles prepared by using copper sulphate and sodium hydroxide as an cationic and anionic sources. From the graph it was observed that, when the lamp was on, the resistance was decreased abruptly. Beneath illumination, the current density hastily elevated and then remained relatively constant, and when the illumination was interrupted, the current density returned to its pre-illumination value.

This increase was predictable due to better crystallinity, which is closer to that of the commercial CuO. Transient peaks occurred due to a large degree of surface electron recombination. Presumably, a more efficient charge transfer occurs for smaller particles that have larger surface contact with the electrolyte. This effect can be further influenced by combining oxides with more conductive additives for better photo-carrier harvesting.

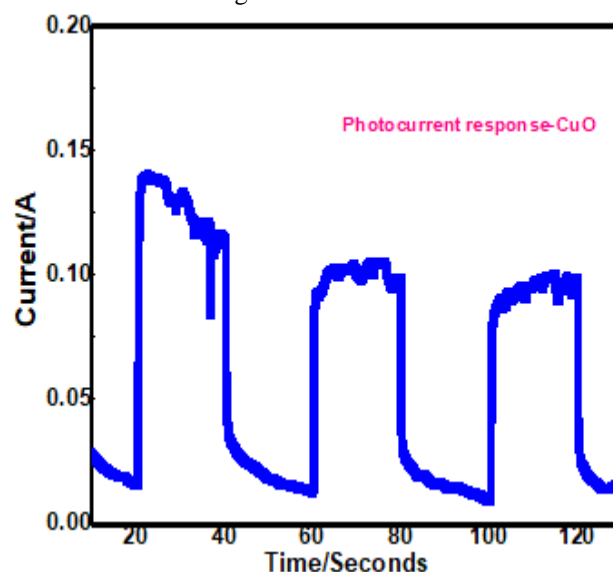


Figure 3: Transient photocurrent response of as-synthesized CuO Nanoparticles

4. Conclusion

In the present work, investigated the CuO nanoparticles were synthesized by using simple precipitation technique. The XRD studies revealed that the as-synthesized CuO nanoparticles were monoclinic structure along with the (002) plane preferential orientation with fine crystalline in nature. HRSEM studies discovered the surface morphology and the nano rod shaped structure of CuO nanoparticles. As compared to the other nanoparticles, CuO that was synthesized through simple precipitation method demonstrated an increase in photocurrent. Consequently from the results, demonstrate that the CuO nanoparticles could be one of the capable candidate of efficient and photoelectrochemical performance.

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