

Synthesis and Characterization of Rice-Grain-Shaped Nanostructured Copper Oxide by a Chemical Method

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Abstract: *The work presented in this article aims to synthesize nanostructured copper oxide (CuO). The nanostructured copper oxide was synthesized by a chemical method, specifically the simple, low-cost co-precipitation method. The objectives of the present work are to synthesize and characterize nanostructured copper oxide using copper nitrate as the precursor. The synthesized nanostructured copper oxide samples were characterized by various methods, such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), ultraviolet-visible (UV-Vis) spectroscopy, and field emission scanning electron microscopy (FESEM) with energy dispersive X-ray spectroscopy (EDS). The XRD and FTIR results confirm the pure form of copper oxide. The crystallite size observed is approximately 15-19 nm, according to the XRD results. The UV-Vis results show absorption peaks below ~450 nm, which also confirms the formation of copper oxide with a band gap of approximately 2.75 - 3 eV. FESEM images reveal a rice-shaped morphology. EDS results reveal the elemental composition of the CuO sample.*

Keywords: CuO nanostructures, chemical method, XRD, FESEM-EDS

1. Introduction

Recently, metal oxide nanostructures have gained research interest, since their exclusive characteristics appear at the nanoscale compared to those of the bulk form [1]. Nanostructured metal oxides possess various functionalities, in addition to enhanced selectivity and reactivity, due to their high density, high surface area-to-volume ratio, and increased reactivity [1-4].

Metal oxides in the nanoscale size range have proven vital uses in various fields, such as fluorescence, optics, chemical, gas and biosensors, catalysts, photovoltaics, biomedicine, fuel cells, etc. The size and shape of nanostructured metal oxides primarily change the surface-dependent properties, thereby affecting their optics, mechanics, electricity, magnetism, and catalytic behavior [5-9]. By controlling the growth and design of nanostructured metal oxides, one can achieve tunable properties, which enhance the activities of these materials and increase their demand in industries [5-7, 10], as well as other fields. Hence, various research efforts have been devoted to manipulating morphologically controlled nanomaterials with uniform size for desired properties [1-3, 11]. In this article, we aim to synthesize nanostructured copper oxide and confirm its structure and morphology using various characterization techniques. Since copper oxide in nanoform is used in different applications, including sensors, photovoltaics, and catalytic applications [12-19].

2. Experimental

Materials

All of the chemicals used were of analytical grade. Copper nitrate hexahydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and NaOH were

purchased from S. D. Fine-Chem Ltd., Mumbai, India, and were used as-received without any further purification.

Synthesis of CuO nanoparticles

Co-precipitation synthesis of copper oxide (CuO) nanostructures was carried out. The reaction solution was prepared by mixing a 0.1 M copper nitrate solution in deionized water. Ammonia solution in distilled water was prepared, and this solution was added dropwise to the copper nitrate solution under vigorous stirring until the pH of the resulting solution became 10-11. The suspension was stirred continuously for two hours at room temperature. The black precipitate in the reaction solution was collected, filtered, washed several times with deionized water, and dried at 90 °C in air. The resulting yield was a white-colored powder with a mass of approximately 0.3 g.

Formation of CuO takes place according to the following reaction:

3. Result and Discussions

3.1 XRD analysis

The XRD pattern of the synthesized CuO powder sample is shown in Figure 1. The largest peaks are observed at the 2θ values of 35.55°, 35.57°, 38.41°, and 38.73°, which correspond to the crystal planes of (0 0 2), (1 1 1), (1 1 1), and (2 0 0), respectively. The other significant Bragg's reflection peaks correspond to the crystal planes (1 1 0), (1 1 2), (2 0 2), (0 2 0), (2 0 2), (1 1 3), (0 2 2), (3 1 1), (1 1 3), (2 2 0), and (3 1 1). The XRD pattern matches the Tenorite, CuO phase with PDF card 90-15822, possessing the space group C 1 2/c 1

(15). The crystallite size, calculated using the Debye-Scherrer formula [$t = (0.9 * \lambda) / (B * \cos \theta)$], is 19 nm.

3.2 FTIR analysis

FTIR spectrum of CuO shows bands at ~414, 597.92, and 498 cm^{-1} that reveal the vibrations of the Cu-O mode, as shown in Figure 2. The bands below 1000 cm^{-1} correspond to metal oxides [19, 20]. The bands at 3300 and 2990 cm^{-1} correspond to O-H stretching due to water content in the sample from the atmosphere, since the powder sample is hygroscopic in

nature. Further, bands at 1500 and 1100 cm^{-1} correspond to O-H bending.

3.3 UV-Vis spectroscopic analysis:

The observations show the UV-Vis spectrum in Figure 3. The graph shows the maximum absorption peaks below 450 nm, which indicates the formation of CuO and Cu_2O nanoparticles. The band gap of the synthesized CuO sample is 1.9 eV.

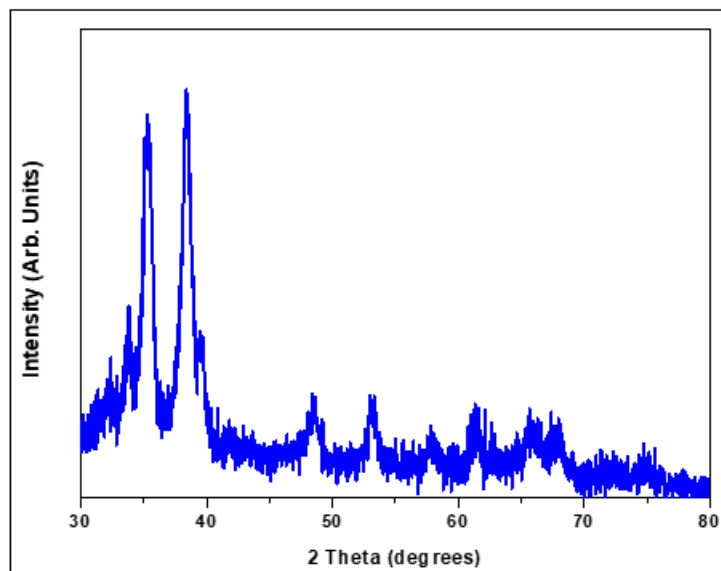
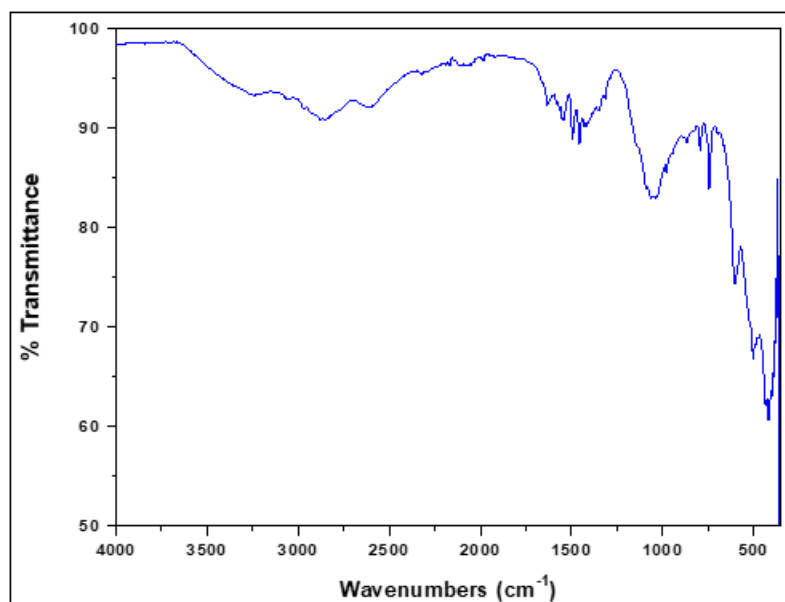


Figure 1: XRD pattern of as synthesized copper oxide powder

3.4 FESEM analysis



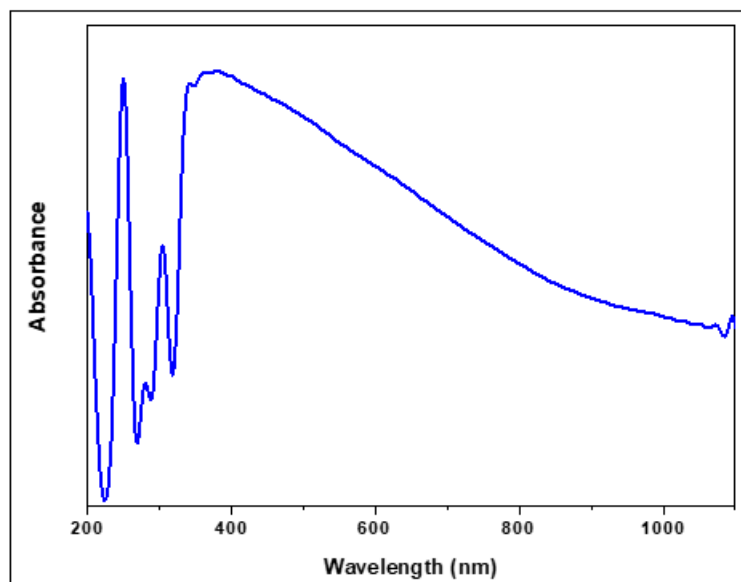


Figure 2: FTIR spectrum of as synthesized copper oxide powder

FESEM image shown in figure 4, is synthesized CuO sample, it shows that the rice like shaped morphology which is not uniform. Some part showing small nanorods having dimensions nearly width 40-50 nm and length nearly 500-600 nm.

4. Conclusions

The nanostructured copper oxide was synthesized by the simple, low-cost co-precipitation method. The XRD and FTIR results confirm the pure form of copper oxide. The crystallite size observed is ~15-19 nm from the XRD results. The UV-Vis results show that the absorption peaks are below ~450 nm, which also confirms the formation of copper oxide with a band gap of ~2.75 – 3 eV. FESEM images show a rice-shaped morphology. EDS results reveal the elemental composition of the CuO sample.

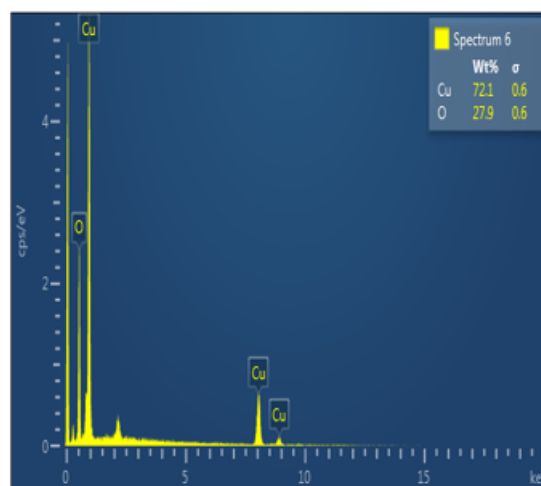


Figure 4: FESEM image and EDS plot of as synthesized copper oxide powder

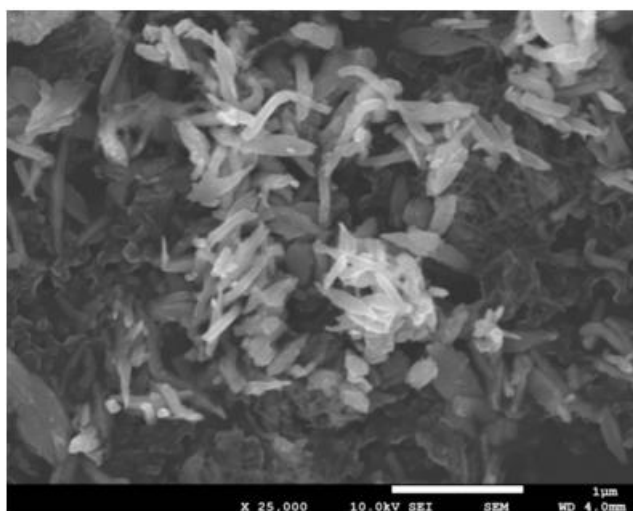


Figure 3: UV-Vis. spectrum of as synthesized copper oxide powder

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