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Short Communication

Template-free solvothermal synthesis of copper oxide nanorods

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ABSTRACT

A simple and efficient approach is reported to surfactant and templates-free synthesis of one-dimensional copper oxide nanostructured material by a solvothermal technique. A uniform and monodisperse copper oxide nanorods are revealed by Transmission Electron Microscopy with the diameter and length of 15 and 90 nm, respectively. X-ray diffraction pattern confirms the monoclinic crystal phase of the copper oxide nanorods with the crystallite/grain size of 15 nm. Downward Raman shift in the vibrational modes with broad peaks infer the quantum size effect of copper oxide nanorods. Formation of monoclinic copper oxide is also confirmed by Fourier Transformation Infrared Spectroscopy.

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1. Introduction

The physical and optical properties of the nanomaterials differ considerably from those of the bulk substances [1]. In the past few years, advancements in manufacturing of nanomaterials have increased dramatically due to their unique physical and chemical features [2]. Recently the synthesis and investigation of zero-dimensional (0D), one-dimensional (1D) and two-dimensional (2D) nanomaterials structure like nanorods and nanowires have drawn great attentions owing to their unique physical, chemical and electronic properties, and potential for their applications in nanodevices [3].

Cupric oxide (CuO), as an important p-type semiconductor metal oxide with a band gap between 1.2 and 1.5 eV, has feasible applications such-as gas sensors [4], solar photovoltaics [5], heterogeneous catalysis [6], lithium ion electrode [7], dye-sensitized solar cells [8], field emission emitters [9], etc. CuO is a unique monoxide compound for both fundamental investigations and practical applications. It has been used as heterogeneous catalysts in many important chemical processes, such as degradation of nitrous oxide, selective catalytic reduction of nitric oxide with ammonia, oxidation of carbon monoxide, hydrocarbon and phenol in supercritical water [10]. CuO nanostructures may be prepared in the form of nanoparticles [11], nanoribbons [12], nanosheets [13], nanoneedles [14], nanorings [15], nanowhiskers [16], nanorod [17], nanotubes [18], nanoleaves [19], nanoflower [20] and nanowires [21]. The interesting shape evaluation of CuO leads to various synthesis methods with different precursors. Among this, synthesis of CuO nanorods has gained great deal of attention due to its high yield in possible solid state devices. However, mechanisms for CuO nanorods formation in aforementioned methods hold the impact on the addition of surfactant and templates in the synthesis process. These surfactants or templates may play a role on the surface of the nanorods which results in poor quality of the products.

In this work, we report surfactant and template free synthesize of CuO nanorods by a simple chemical route method. This method requires neither complex apparatus nor long synthesis times.

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2. Materials and methods

The precursor materials Copper chloride ($CuCl_2 \cdot 2H_2O$) and Sodium hydroxide (NaOH), were purchased from Merck and Aldrich. The chemicals were analytical reagent grade and used in the experiment without further purification treatment.

In a typical synthesis, 0.5 mol (34.28 gm) of Copper chloride ($CuCl_2 \cdot 2H_2O$) was dissolved in 100 ml deionised water (H_2O), using a magnetic stirrer. This solution was stirred for 30 min until the $CuCl_2$ dissolved completely. Then, Sodium hydroxide (NaOH) solution was added dropwise into the $CuCl_2$ solution, under constant stirring. During this process, the reaction mixture forms a bluish green gel and then changed into dark brown Copper (II) hydroxide ($Cu(OH)_2$) precipitate completely. The precipitate of Cu $(OH)_2$ was washed with distilled water several times and then centrifuged to remove the native impurities in the product. Finally, the $Cu(OH)_2$ precipitate was annealed at 350 °C for around 2 h in air until $Cu(OH)_2$ was turned into CuO completely.

2.1. Characterizations

Structural analysis of the sample was carried out using a SHIMADZU 600 X-ray diffractometer with Kα radiation with the scanning angles of 20–90°. The 1D nature of the CuO nanorods was revealed using TECNAI Transmission Electron Microscopy. Raman studies were carried out using a Lab RAM HR 800 micro Raman spectrometer with 514.12 nm laser source. Optical analysis was carried out using a SHIMADZU IR-Prestige 21 Fourier transformation infrared spectrometer.

3. Results and discussion

3.1. X-ray diffraction analysis of CuO nanorods

Fig. 1 shows the X ray diffraction (XRD) pattern of synthesized CuO nanorods. All the diffraction peaks confirm the monoclinic phase of CuO, and are in good agreement with the "JCPDS"card no.80-1916. The nanorods obtained consist good quality pure CuO phases.

The average crystallite diameter of the CuO nanorod calculated using Debye–Scherrer equation was about 15 nm. The sharp and intense peaks in the XRD pattern of the product indicate good crystallinity of CuO nanorods. The obtained lattice parameters are (a=4.692 Å, b= 3.428 Å, c=5.137 Å and β=99°24′ with volume cell of 81.50 $Å^3$) consistent with the respective JCPDS card.

3.2. Transmission electron microscopy analysis of CuO nanorods

The structures and shapes were preserved even after the CuO structure was dispersed in ethanol by ultrasonic vibration for 30 min before being deposited on a carbon-coated copper grid for the Transmission Electron Microscopy (TEM) observations. Fig. 2 and Fig. 3(a) and (b) show TEM images of CuO nanorods with different magnifications.

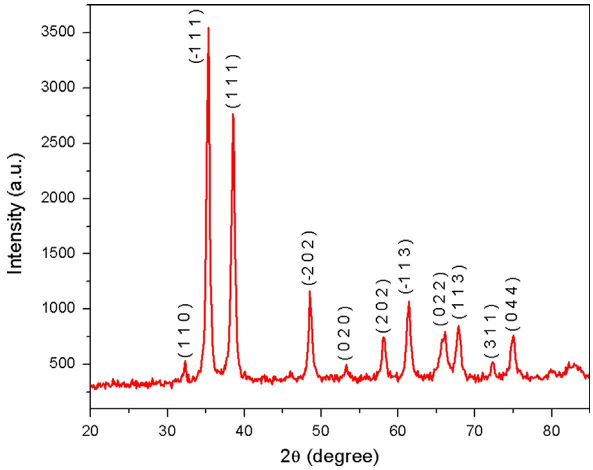

Fig. 1. XRD spectrum of copper oxide nanorods.

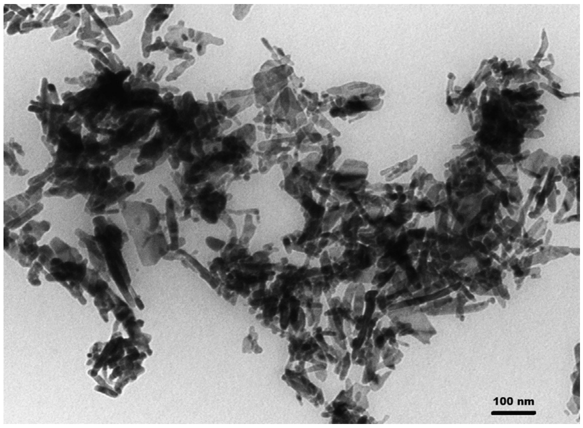

Fig. 2. TEM images of copper oxide nanorods.

A relatively straight rod-like shape of smooth surfaces is clearly displayed in Fig. 2 and Fig. 3 (a). The CuO nanorods are randomly oriented and this is a tradeoff for not using templates for preparation. An examination of single nanorod view of Fig. 3(b) shows the average length of 80–150 nm with the diameter of 12–17 nm.

3.3. Laser Raman analysis of CuO nanorods

Raman spectroscopy has been widely used as a sensitive tool to investigate the microstructure, namely the local atomic arrangement and vibration of the nano-sized materials. One of the effective ways to investigate quantum confinement effects are such as the dramatic down shift and broadening of Raman peaks with broad vibrational modes [22]. CuO nanorods with the monoclinic structure belong to the space group symmetry of C^6_{2h}, which contains two molecular units per primitive cell. Thus, there are 12 zone-center optical phonon normal vibrational modes $\Gamma_{RA}=4A_u+5B_u+A_g+2B_g$ including six infrared active modes ($3A_u+3B_u$), three acoustic modes (A_u+2B_u), and three Raman active modes (A_g+2B_g). Three

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