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Synthesis and characterization of CdO nanocrystalline structure by mechanochemical method

A. Tadjarodi *, M. Imani

Research Laboratory of Inorganic Materials Synthesis, Department of Chemistry, Iran University of Science and Technology, 16846-13114, Tehran, Iran

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ABSTRACT

Cadmium oxide nanostructure was synthesized by calcining the obtained precursor of a mechanochemical reaction. The milling was carried out with cadmium nitrate tetrahydrate and acetamide reactants without any additives at room temperature. Resulting precursor was calcined at 450 °C for 2 h in a furnace. As a result of heating, the organic section of precursor was removed and cadmium oxide nanostructure was produced. The obtained compound from the mechanical milling (MM) technique possesses a cubic crystalline structure at nanoscale. XRD studies indicate that the obtained CdO has a cubic phase. Also, SEM and TEM images showed that the resulting material is composed of nanoparticles with the average diameter of 41 nm. The average size and standard deviation were calculated using a Microstructure Measurement program and a Minitab statistical software.

1. Introduction

Recently, researchers have focused on the synthesis of nano-sized semiconductor metal oxides because of their optical and electrical properties, which has led to new applications in the industry [1]. Nanocrystalline cadmium oxide (CdO) is an important n-type semiconductor metal oxide with a direct band gap of 2.2–2.7 eV and an indirect band gap of 1.36–1.98 eV [2]. Different values for band gap have been reported in the literatures that can be the result of a lattice's defects originated from different preparation conditions [3]. CdO with different structures has been used for solar cells, photo transistors, transparent electrodes, catalysts, gas sensors, optoelectronic devices, anode materials for lithium ion batteries and photodiodes [2–9].

Numerous methods have been introduced to prepare CdO in nanoscale, including vapour transport, chemical vapour deposition (CVD), sol-gel, chemical bath deposition (CBD), laser ablation, spray pyrolysis and solvothermal/hydrothermal methods and also mechanochemical process with various terms such as high-energy ball milling–annealing, mechanical alloying (MA), mechanical milling (MM) or mechanical grinding (MG) and other ways that have been reported in the literatures [2–16]. Nowadays, utilization of the mechanochemical process in comparison with other methods is increasing because it is an effective, useful, low-cost and a simple technique to synthesize different structures of nanomaterials [17]. This method has been developed by Benjamin and his coworkers in 1960s as milling mixing for the first time. Then, Mac-Queen, et al. described its industrial application [18]. A lot of research in the field of preparation and application of this material has remained to be done.

In this study, nanocrystalline metal oxide powder of CdO was prepared by mechanochemical process. The product was characterized by FT-IR, XRD, SEM and TEM techniques.

2. Experimental

2.1. Materials of synthesis

All of the chemical used in this work were purchased from Merck and used without further purification.

2.2. Preparation

$Cd(NO_3)_2{\cdot}4H_2O$ (2.05 g) and CH_3CONH_2(0.51 g) were mixed together with a molar ratio of 3:4 and then, they were put in a stainless steel cylinder (10 mL) with two small balls of 10 mm diameter by utilizing a mass ratio of 8:1 ball-to-powder. Milling was carried out using Mixer Mill (Retsch MM-400) of grinding system for 30 min with a rate of 30 Hz (1800 rpm) at room temperature and the resulting precursor was calcined at 450 °C for 2 h.

2.3. Characterization

The powder X-ray diffraction (PXRD) measurements were carried out by a JEOL diffractometer with monochromatized Cu Kα radiation (λ = 1.5418 Å). Fourier transform infrared (FT-IR) spectra were recorded on a Shimadzu-8400S spectrometer in the range of 400–4000 cm^{-1} using KBr pellets. Scanning electron microscopy

* Corresponding author. Tel.: +98 21 77240516; fax: +98 21 77491204.
E-mail address: tajarodi@iust.ac.ir (A. Tadjarodi).

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