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Microwave-assisted synthesis of $Cd(OH)_2$/CdO nanorods: Effect of irradiation time

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ABSTRACT

A simple method to synthesize cadmium hydroxide/oxide nanorods by a wet chemical route assisted by microwave irradiation in the presence of a bio-template is presented. The obtained products at different irradiation times have been characterized by X-ray diffraction (XRD), Transmission electron microscope (TEM), Diffuse Reflectance Spectroscopy (DRS), and Fourier transform infra-red spectroscopy (FT-IR). XRD analysis showed the presence of crystalline $Cd(OH)_2$ and CdO phases, in the monoclinic and cubic structure, respectively. The albumen biotemplate addressed the growth of crystalline $Cd(OH)_2$ and CdO along the c axis, generating nanorods with diameter approximately of 20–50 nm and length up to 500 nm, assembled to form elongated hierarchical nanostructures of some micrometers. The optical and electrical properties of these nanostructures were also investigated.

1. Introduction

The synthesis of nanostructured metal hydroxides/oxides is an important step in the preparation of various semiconductor materials. Nanostructures (nanoparticles, nanowires, and nanobelts) of metal hydroxides/oxides have attracted much attention because of their size-induced novel properties for developing a variety of optoelectronic devices [1].

Metal hydroxides are generally regarded as precursors during the synthesis of metal oxides, but various metal hydroxides like $Ni(OH)_2$, $Cu(OH)_2$, Mg $(OH)_2$ and $Cd(OH)_2$ show special physical characteristics with interesting applicative properties [2–5]. Previously, we investigated the synthesis of cadmium hydroxide/oxide materials for optical and sensing applications [6–8]. The formation of nanotubes, nanorods, nanowires and nanobelts of cadmium hydroxide is highly attractive due to their unique electronic and optical properties [9–11]. $Cd(OH)_2$ is a wide band gap (3.2 eV) semiconductor material [12]. The wide band gap $Cd(OH)_2$ can be used in different applications such as solar cells, phototransistors, diodes, transparent electrodes, sensors etc. [13]. CdO is an n-type semiconductor with band gap of ~2.5 eV, showing lower resistivity. Due to its high electrical conductivity and optical transmittance in the visible region of solar spectrum, it has great potential for advanced applications (flat panel display, organic light emitting diodes, gas sensors etc.). So, for example, cadmium hydroxides/oxides nanostructures have been studied considerably in the past decade for developing highly sensitive sensor devices [6].

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The optical and electrical property of cadmium hydroxide/oxide depends on their morphological and microstructural characteristics such as stoichiometry, size and shape of the particles. Then, the procedure route is critical in the synthesis of these materials [14–17]. Therefore, seeking a simple approach for low-cost, lower-temperature, large-scales, controlled growth of metal hydroxides/oxides nanostructures is highly desired.

This work is focused on the use of a simple wet chemical approach using a biotemplate (albumen) and assisted by microwave irradiation for different time (5, 10 and 15 min) with aim to prepare $Cd(OH)_2$/CdO nanomaterials [18–22]. The morphology and microstructure of the obtained materials were investigated by XRD, FT-IR, TEM, UV-DRS. The optical and electrical properties are also reported.

2. Experimental procedure

2.1. Materials

Cadmium chloride, $Cd(Cl)_2$ and ammonia, (NH_4OH) were supplied from (Merk, 98%) Mumbai, India. All the chemicals were of analytical grade and used as received without further purification. The albumen, extracted by white part of egg, was used for the synthesis. Double distilled water was used throught the experiments.

2.2. Synthesis

A 0.1 M solution of $Cd(Cl)_2$ in deionized water was prepared. The pH of the solution was maintained as 8 using ammonia diluted with water. The 5 ml of egg solution was added drop wise in above solution at constant stirring. The resulting precipitate was separated and washed with water more than five times until the removal of chlorine ions and finally with ethanol. The precipitate was separated in three portion, which were irradiated with microwave radiation frequency (2.45 GHZ, power up to 1 KW) in a microwave oven for 5, 10 and 15 min, respectively. These samples were named according to the irradiation time, Sample A for 5 min, Sample B for 10 min, Sample C for 15 min (See Table 1).

2.3. Characterization

The samples were characterized by different techniques as follows. X-Ray diffraction (XRD) data were recorded with the help of Bruker (AXS D8 Advance) diffractometer with CuK_α radiation ($\lambda = 1.5406$ Å) operating at 40 kV and 30 mA. The crystallites average size was determined by the Scherrer equation (Eq. (1)).

$$D_{hkl} = \frac{0.9\lambda}{\beta_{hkl}\cos(\theta_{hkl})} \tag{1}$$

where λ is the X-ray wavelength, θ_{hkl} is the Bragg diffraction angle, and β_{hkl} is the full width at half-maximum (FWHM) of the main peak in the XRD pattern. Transmission electron microscopy (TEM) and selected-area electron diffraction (SAED) were recorded on a Philips (Mod. CM 200), electron microscope with an acceleration voltage of 80 kV. Fourier transform infrared spectroscopy (FT-IR) spectra were recorded using a Bio Rad spectrometer (Mod. FTS-7). Diffuse reflectance spectroscopy (DRS) spectra were recorded on a Perkin Elmer UV–visible DRS spectrophotometer. The electrical resistance measurements were carried out using a Keithley electrometer (Mod. 6517B).

3. Results and discussion

3.1. Morphological and microstructural analysis

Fig. 1(a–c) shows the X-ray diffraction pattern of microwave irradiated samples for 5, 10 and 15 min. XRD patterns of all products obtained show diffraction peaks corresponding to the (020), (011), (200), (040), $(\bar{1}41)$,$(\bar{2}31)$,(051), $(\bar{3}21)$,$(\bar{2}51)$ and

Table 1
Main characteristics of the sample investigated.

Sample Code	Microwave treatment	Crystalline phase	Average crystallite size, by XRD(nm)	Lattice parameter (Å)
Sample A	5 min	$Cd(OH)_2$ (Monoclinic) & CdO (Cubic)	26 & 23	a = 5.670, b = 10.25, c = 3.410 & a = 4.687
Sample B	10 min	$Cd(OH)_2$ (Monoclinic)&CdO (Cubic)	22 & 19	a = 5.671, b = 10.24, c = 3.411 & a = 4.686
Sample C	15 min	$Cd(OH)_2$ (Monoclinic) & CdO (Cubic)	32 & 29	a = 5.671, b = 10.25, c = 3.410 & a = 4.688