



Controllable synthesis and growth mechanism of α -Co(OH)₂ nanorods and nanoplates by a facile solution-phase route

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ARTICLE INFO

Article history:

Received 14 August 2011

Received in revised form

11 October 2011

Accepted 18 October 2011

Available online 25 October 2011

Keywords:

Inorganic materials

Nanostructured materials

Chemical synthesis

Crystal structure

ABSTRACT

A facile chemical precipitation route has been developed to control synthesis of α -cobalt hydroxide nanostructures with rod-like and plate-like morphologies. The α -Co(OH)₂ nanorods were achieved in large quantity when the experiments were carried out in the presence of a suitable shape-controlling reagent polyvinyl pyrrolidone (PVP), while the α -Co(OH)₂ nanoplates were obtained when the experiments were conducted in the absence of PVP, whilst keeping other experimental conditions constant. The chemical composition and morphologies of the as-prepared α -Co(OH)₂ nanoparticles were characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The effect of polymer PVP on the morphologies of α -Co(OH)₂ nanoparticles were discussed in detail. The results indicated that PVP played a key role for the formation of α -Co(OH)₂ nanorods. The growth mechanism of the as-synthesized nanorods and nanoplates were discussed in detail based on the experimental results. A possible growth mechanism has been proposed to illustrate the growth of α -Co(OH)₂ nanorods.

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1. Introduction

Recently, there are great growing interests for the controllable synthesis of low-dimensional nanoscale materials with well-controlled morphologies due to their novel optical, electronic, magnetic, and catalytic properties and potential applications in the fields of photonic, electronic magnetic devices and catalysts [1]. Much effort has therefore been focused on the design, synthesis, and characterization of nanoscale materials with different morphologies such as nanocubes, nanorods, nanowires, nanobelts, and nanotubes [2–7]. Till date, many methods have been employed to synthesize nanoscale materials such as nanoparticles, one-dimensional (1D) nanostructures and two-dimensional (2D) nanostructures. Among these synthetic methods, both hard and soft template methods are generally used to synthesize nanostructures with well-controlled shapes. In case of hard template method, the alumina, silica, block polymer, mica, and membranes are usually used as templates for the direct growth of nanostructures [8–10]. While in soft template process, surfactants are generally applied to stabilize the surface of nanonuclei and kinetically control the growth rates of various facets of nuclei, leading to the formation of nanostructures with well-controllable morphologies [11–13].

In recent years, cobalt hydroxide has attracted increasing attention because of its unique electric and catalytic properties for electrochemical, magnetic, and catalytic applications. It is well-known that cobalt hydroxide, a divalent transition-metal hydroxide, crystallizes in two polymorphs, the metastable α -type and the thermodynamically stable β -type [14,15]. The previous research works have reported that the thermodynamically stable β -type cobalt hydroxide is a stoichiometric phase of the composition Co(OH)₂ with brucite-like structure and consists of a hexagonal packing of hydroxyl ions with Co(II) occupying alternate rows of octahedral sites [16,17], while the metastable α -type cobalt hydroxide is reported to be isostructural with hydrotalcite-like compounds that consist of positively charged Co(OH)_{2–x}(H₂O)_x layers and charge balancing anions in the interlayer gallery [18–21]. It has been reported that the α -type cobalt hydroxide shows superior electrochemical properties as compared to its β -type counterpart because of its poorly or turbostratically crystallized structure [15]. In addition, the α -type cobalt hydroxide intercalated organic anions show long-range magnetic ordering and are regarded as new candidates for organic magnetic materials [22,23]. The novel electrochemical and magnetic properties of α -type cobalt hydroxide triggered the recent great interest in shape-controlled synthesis of α -type cobalt hydroxide by the chemical or electrochemical process. However, it is difficult to synthesize α -type cobalt hydroxide because it is metastable and is easily transformed into the β -type cobalt hydroxide during the reaction or aging. Although few methods have been developed to

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synthesize α -type cobalt hydroxide nanostructures with mainly plate-like morphologies [24–26], there are a few reports on the synthesis of α -type cobalt hydroxide with rod-like morphology to the best of our knowledge. In the present work, we demonstrate a facile chemical precipitation route for controllable synthesis of α -type cobalt hydroxide nanorods and nanoplates for first time. The growth mechanism of nanorods and nanoplates were discussed in detail on the basis of experimental results and analysis. A possible growth mechanism has been proposed to illustrate the growth of α -Co(OH)₂ nanorods.

2. Experimental procedure

All of the chemical reagents used in the experiments were of analytical grade as received without further purification. In a typical synthetic procedure of α -Co(OH)₂ nanorods, CoCl₂·6H₂O (0.48 g) and HMT (1.68 g) were dissolved in 200 mL of a 9:1 mixture of deionized water and ethanol to give the final concentrations of 10 and 60 mM, respectively. Then 0.20 g of polyvinyl pyrrolidone (PVP) ((K30, molecular weight of 58 000) was dissolved into the above solution. The reaction solution was then heated at 90 °C under magnetic stirring. After being heated for 1 h, a suspension containing green particles were resulted. The solid product was filtered and washed with deionized water and anhydrous ethanol several times, and finally air-dried at room temperature.

The synthetic procedure for α -Co(OH)₂ nanoplates is same as the above procedure for the synthesis of α -Co(OH)₂ nanorods, but no PVP was added in the whole experimental process.

The powder XRD analysis was performed using a Rigaku (Japan) Dmax X-ray diffractometer with graphite monochromatized CuK α radiation ($\lambda=0.154178$ nm), employing a scanning rate of $0.02^\circ \text{ s}^{-1}$ in the 2θ range from 10° to 90° . Transmission electron microscopy (TEM) images were taken with a Hitachi-7650 transmission electron microscope, with an accelerating voltage of 80 kV.

3. Results and discussion

The crystallinity and chemical composition of the as-prepared α -Co(OH)₂ nanorods was first characterized by XRD. Fig. 1 shows typical XRD pattern of α -Co(OH)₂ nanorods obtained by

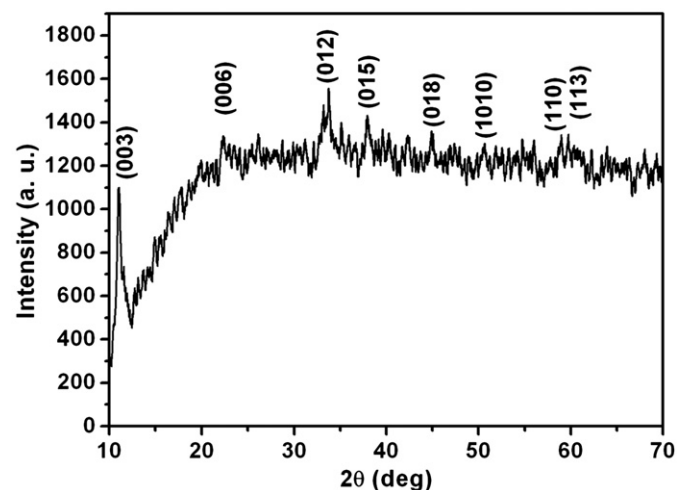


Fig. 1. A typical XRD pattern of α -Co(OH)₂ nanorods obtained by precipitation of CoCl₂ aqueous solution with HMT in the presence of shape-controlled reagent PVP at 90 °C for 1 h.

precipitation of CoCl₂ aqueous solution with HMT in the presence of PVP at 90 °C for 1 h. The peak assignment is made with reference to the previous report about the crystal structure of α -Co(OH)₂ [24–26]. According to the previous published works, five characteristic peaks located at 10.97° , 22.34° , 33.69° , 37.96° , and 58.94° that attributed to (003), (006), (012), (015), and (110) crystalline planes of α -Co(OH)₂ were observed. It was reported that typical low-crystalline α -type cobalt hydroxides generally showed broadened peaks of (003), (006), (100), and (110) planes in the XRD patterns [24]. In contrast, almost all the characteristic peaks of α -Co(OH)₂ are observed on the XRD pattern of the precipitates in the present study.

The morphology and size of the as-prepared α -Co(OH)₂ nanorods have been characterized by TEM. Fig. 2 shows typical TEM images of α -Co(OH)₂ nanorods prepared by a facile chemical precipitation route, in which precipitation of CoCl₂ was used as Co²⁺ source, HMT was used as precipitation reagent, and PVP was used as structure-directing reagent. The low-magnification TEM image (Fig. 2a) shows that the as-prepared α -Co(OH)₂ nanoparticles exhibit a rod-like morphology in large quantity. Medium-magnification TEM image clearly indicates that the as-prepared α -Co(OH)₂ nanoparticles have perfect rod-like shape with an average diameter of about 10 nm and length of up to 1000 nm

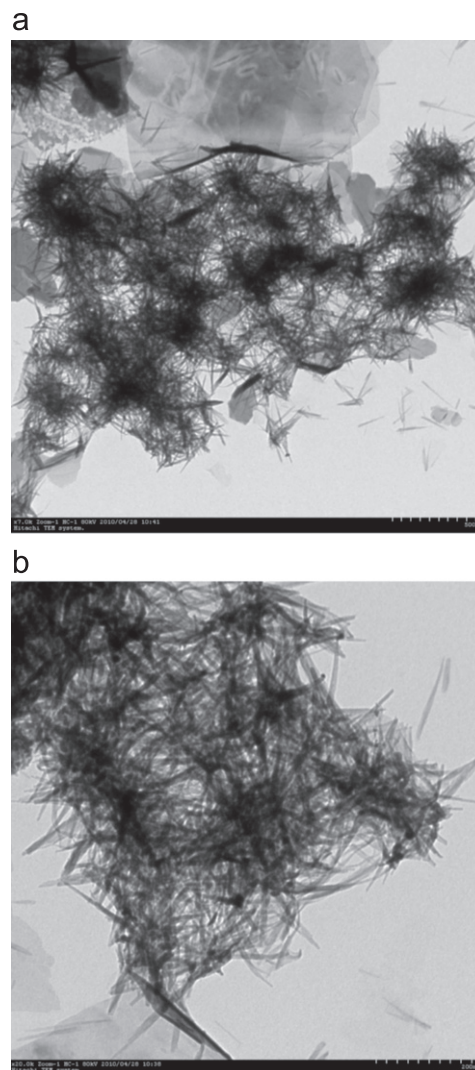


Fig. 2. Typical TEM images of α -Co(OH)₂ nanorods obtained by precipitation of CoCl₂ aqueous solution with HMT in the presence of shape-controlled reagent PVP at 90 °C for 1 h.

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