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Gelatin-assisted green synthesis of bismuth sulfide nanorods under microwave irradiation

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ABSTRACT

Bismuth sulfide (Bi_2S_3) nanorods were synthesized by a gelatin-assisted green solution process under microwave irradiation. The crystal phase, morphology and optical properties of the as-synthesized Bi_2S_3 nanorods were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), UV–vis diffuse reflection spectroscopy (UV–vis DRS) and photoluminescence (PL) spectroscopy. On the basis of the morphological evolution processes, the growth mechanism of the as-synthesized Bi_2S_3 nanorods was discussed. The as-synthesized Bi_2S_3 nanorods exhibited a strong excitonic absorbance in UV–vis DRS and a strong emission peak in PL spectrum. The gelatin-assisted synthesis approach under microwave irradiation is green, facile and efficient thus promoting large-scale production and application of Bi_2S_3 nanorods in photoelectric nanodevice and other fields.

1. Introduction

As an important member of the metal chalcogenides $A_2^VB_3^{VI}$ (A=As, Sb, Bi; B=S, Se, and Te) nanomaterials, bismuth sulfide (Bi_2S_3) has received intense attention of many researchers because of its various potential applications in extensive areas of science and technology such as photovoltaic devices, dye-sensitized solar cells, photocatalysis, thermoelectric devices, lithium-ion batteries, and so on [1–4]. A lot of research have been focused on the synthesis of Bi_2S_3 nanostructures, and a series of different dimensionalities and morphologies such as nanoparticles [5], nanorods [2,6,7], nanowires [8], nanosheets [9], nanotubes [1] and microspheres [3,4,10–12], have been successfully synthesized by various methods. More importantly, the Bi_2S_3 nanostructures exhibit distinctive morphology-dependent physicochemical properties. Therefore, the design and synthesis of the Bi_2S_3 nanostructures are full of challenges and opportunities.

Recently, the synthesis of nanostructures assisted by biomolecules such as amino acids [13], β-cyclodextrin [14], glucose [15], starch [16], alginate [17] and gelatin [18,19], represents features of sustainable and efficient in morphology-controlled synthesis of various functional nanomaterials. Among various biomolecules, gelatin is a green and low-cost biomolecule derived from animal skin and bone, containing a large number of organic functional groups, such as amino, carboxylic and so on [19]. Gelatin biomolecules could significantly influence the assembly and morphology of inorganic nanostructures due to the complexation of their polar groups with metal ions. For instance, Stroyuk et al. prepared mesoporous TiO_2 with higher photocatalytic activity in air treatment using gelatin as a template [18]. Fang et al. reported that gelatin-assisted hydrothermal synthesis of single crystalline ZnO nanostars [19]. Nevertheless, to the best of our knowledge, little work has been made to the controllable and facile synthesis of Bi_2S_3 nanostructures assisted by gelatin biomolecules.

In this paper, we report a green and facile gelatin-assisted synthesis of Bi_2S_3 nanorods under microwave irradiation. The crystal phase, morphology, growth mechanism, UV–vis diffuse reflection spectrum (UV–vis DRS) and photoluminescence (PL) spectrum of the Bi_2S_3 nanorods were investigated.

2. Experimental

All chemicals were of analytical grade except gelatin, which was of chemically pure reagent. In a typical synthesis, 0.61 g of $Bi(NO_3)_3$ and 0.10 g of gelatin were dissolved in 50 mL deionized water under continuous stirring. The mixture was stirred for 10 min at 60 °C, 0.95 g of thiourea was added in the above mixture. Subsequently, the sample was stirred for 20 min, and then was transferred into a 250 ml round flask and heated by microwave irradiation in a microwave oven (800 W, 100 °C and 2450 MHz) with a refluxing apparatus for 30 min. The resulting Bi_2S_3 nanorods were collected by centrifugation, washed using deionized water and dried 12 h at 80 °C.

The X-ray powder diffraction (XRD) pattern of the sample was recorded using a PANalytical X'Pert PRO diffractometer with monochromatized CuKα radiation (λ=0.154056 nm). The operating

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voltage and current were 40 kV and 40 mA, respectively. Scanning electron microscopy (SEM) images of the samples were taken with a Hitachi S3400 microscope operated at an acceleration voltage of 5.00 kV. Transmission electron microscopy (TEM) was performed on a FEI Tecnai G2 F20 S-TWIN microscope under an acceleration voltage of 200 kV. UV–vis DRS of the sample was obtained on a UV–visible spectrophotometer (Shimadzu UV-2550) equipped with an integrating sphere attachment and $BaSO_4$ as reference. Room temperature PL measurement was carried out with a luminescence spectrometer (Hitachi F2500).

3. Results and discussion

Fig. 1 shows the crystal phase and the morphology of the product. As shown in Fig. 1a, the XRD pattern of the product can be indexed as a pure orthorhombic phase of Bi_2S_3 (JCPDS no. 17-0320). No diffraction peaks of impurities appeared in the XRD pattern. The SEM image of the product in Fig. 1b shows that the product consists of a large number of nanorods with a length ranging from several hundred nanometers to several microns. The TEM image of the product in Fig. 1c exhibits the nanorods have a diameter in the range of about 20–50 nm. The HRTEM image in Fig. 1d reveals that the lattice spacing of the nanorods is about 0.397 nm. The value can correspond to the (220) plane lattice parameter of orthorhombic Bi_2S_3.

To understand the role of gelatin in the formation of Bi_2S_3 nanorods, we examined morphological evolution with a different amount of gelatin. Fig. 2 shows TEM images of the different products. The product in absence of gelatin consists of irregular and micron-sized structure as shown in Fig. 2a. Interestingly, when 0.04 g of gelatin was added to the reaction system, the product was converted into densely packed nanorods with diameters of about 80–100 nm and lengths of about 500–1000 nm as

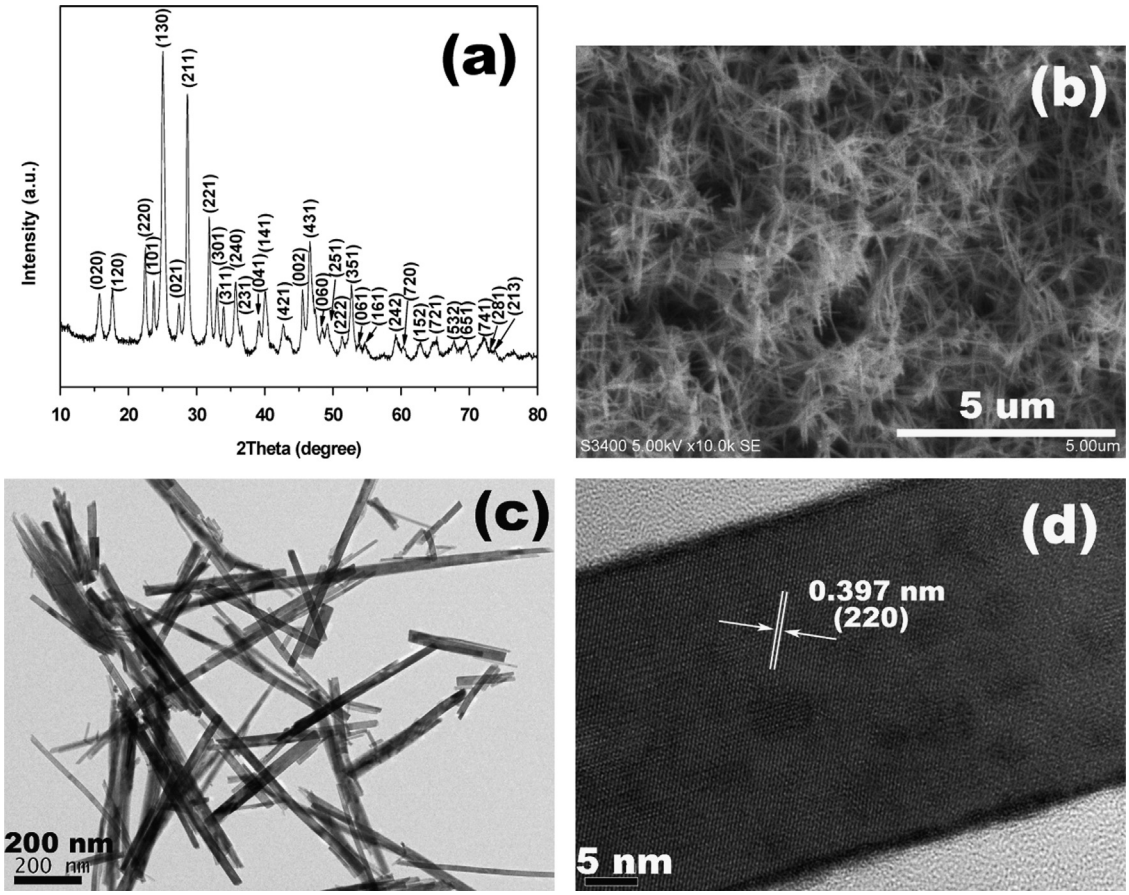

Fig. 1. (a) XRD pattern, (b) SEM image, (c) TEM image and (d) HRTEM image of the product.

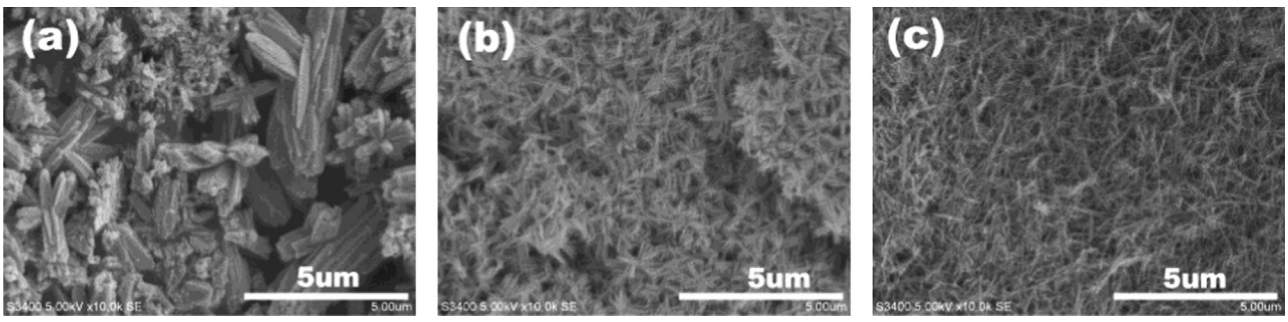

Fig. 2. TEM images of the products synthesized with a different amount of gelatin: (a) 0 g, (b) 0.04 g and (c) 0.07 g.