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Short communication

Hydrothermal synthesis of hexagonal magnesium hydroxide nanoflakes

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ABSTRACT

Hexagonal magnesium hydroxide ($Mg(OH)_2$) nanoflakes were successfully synthesized via hydrothermal method in the presence of the surfactant polyethylene glycol 20,000 (PEG-20,000). Results show that PEG-20,000 plays an important role in the formation of this kind of nanostructure. The composition, morphologies and structure of the $Mg(OH)_2$ nanoflakes were characterized by X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM), high-resolution transmission electron microscopy (HRTEM), and selected area electron diffraction (SAED). The SAED patterns taken from the different positions on a single hexagonal $Mg(OH)_2$ nanoflake show different crystalline structures. The structure of the nanoflakes are polycrystalline and the probable formation mechanism of $Mg(OH)_2$ nanoflakes is discussed. Brunauer–Emmett–Teller (BET) analysis were performed to investigate the porous structure and surface area of the as-obtained nanoflakes.

1. Introduction

During the past few decades, low-dimensional nanostructured materials have attracted considerable attention because of their unique electronic, optical, catalytic, sensory, and magnetic properties [1–13]. Studies have shown that the properties of nanomaterials and micro-materials are highly size- and shape-dependent, and therefore it is extremely important to be able to properly control the size and morphology of these materials. Essentially, the hydrothermal synthesis in the presence of different surfactants or combined with a calcination process is a useful tool for the synthesis of low-dimensional nanomaterials due to its distinct advantages such as its single-step process at low temperatures, wide selection of composition, and morphological control [14–27].

$Mg(OH)_2$ is a metal hydrate, which is innocuous, it has no smell, a low density, and it is noncombustible. $Mg(OH)_2$ has many applications due to its outstanding physical and chemical properties. For example, $Mg(OH)_2$ can be used as a catalyst, catalysts support, a sorbent for chemical and destructive adsorption of various pollutants, and it can also be used in fire-retardant materials [26–35]. Although the synthesis of $Mg(OH)_2$ has been studied by many researchers [30–52], there is no report on the synthesis of a multi-selected area, polycrystalline structure yet.

Here, we report the hydrothermal synthesis of regular, hexagonal $Mg(OH)_2$ nanoflakes with a thickness of about ~10 nm and a lateral size of about ~100 nm in the presence of PEG-20,000. Results show that PEG-20,000 plays an important role in the formation of this kind of nanostructure.

2. Experimental

All the reagents used in this experiment were analytically pure and were received from commercial sources. All the chemicals were used without further purification. Water used in this study was distilled and deionized.

2.1. Synthesis

In a typical synthesis, 1.0 mmol of hydrated magnesium nitrate ($Mg(NO_3)_2{\cdot}6H_2O$) and 5×10^{-3} mmol of polyethylene glycol 20,000 (PEG-20,000) were each dissolved in 30 mL of deionized water. Then the aqueous solution of PEG-20,000 was added dropwise to the aqueous $Mg(NO_3)_2$ solution under constant stirring. The mixture was stirred until a clear and homogeneous

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solution formed, and 1 mL of 1.0 M NaOH was added to this mixture. Then the as-obtained suspension was transferred to an 80 mL Teflon-lined autoclave, which was sealed and maintained at 160 °C for 12 h, and then cooled to room temperature in air. After the autoclave was cooled to room temperature, the resulting white precipitate was collected and washed several times with distilled water and absolute ethanol. The yield of $Mg(OH)_2$ hexagonal nanoflakes was up to almost 100%.

2.2. Characterization

Hexagonal $Mg(OH)_2$ nanoflakes were characterized using X-ray powder diffraction (XRD, Shimadzu XRD-6000, Cu Kα radiation (λ = 1.54 Å)), field-emission scanning electron microscopy (FE-SEM, Hitachi S-4800) with an accelerating voltage of 10 kV, and high-resolution transmission electron microscopy (HRTEM JEOL 2010F) with an accelerating voltage of 200 kV. XRD samples were prepared by putting and pressing 10 mg $Mg(OH)_2$ samples on the test vessel. FE-SEM samples were prepared by putting 1 mg $Mg(OH)_2$ on the test vessel and covering it with a conductive fabric. These samples were then conductively coated with gold by sputtering for 60 s to minimize charging effects under FE-SEM imaging. HRTEM samples were prepared by depositing a drop of the suspended nanoflakes in dilute alcohol onto the surface of a 300 mesh copper grid, coated with a lacey carbon film and dried in a vacuum chamber for 1 h. Prior to deposition, solutions containing samples of the hexagonal $Mg(OH)_2$ nanoflakes were sonicated for 2 min to ensure adequate dispersion of the hexagonal nanoflakes. The specific surface area, average pore diameter, pore volume of the homemade and irregular $Mg(OH)_2$ were determined on a constant volume adsorption apparatus (CHEMBET-3000) by the N_2-BET method at liquid nitrogen temperature.

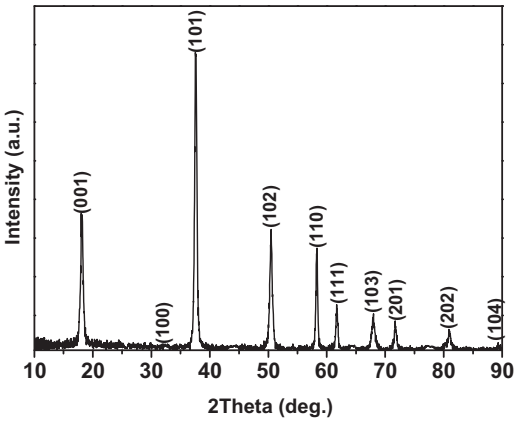

Fig. 1. XRD patterns of hexagonal $Mg(OH)_2$ nanoflakes.

3. Results and discussion

3.1. X-ray diffraction

The composition and purity of the as-synthesized $Mg(OH)_2$ hexagonal nanoflakes were examined by X-ray diffraction (Fig. 1).

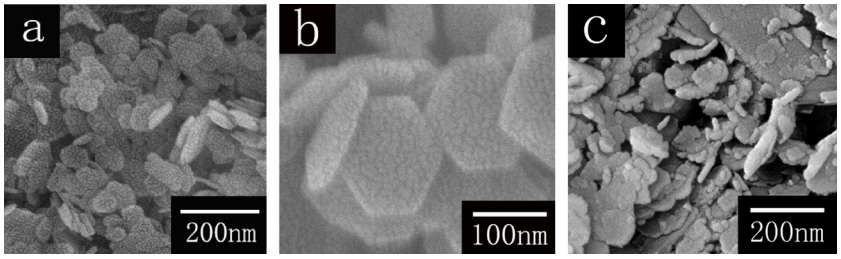

Fig. 2. FE-SEM images of $Mg(OH)_2$ samples. (a) Low-magnification of hexagonal $Mg(OH)_2$ nanoflakes. (b) Higher magnification of hexagonal $Mg(OH)_2$ nanoflakes. (c) Irregular $Mg(OH)_2$ flakes synthesized without PEG-20000.

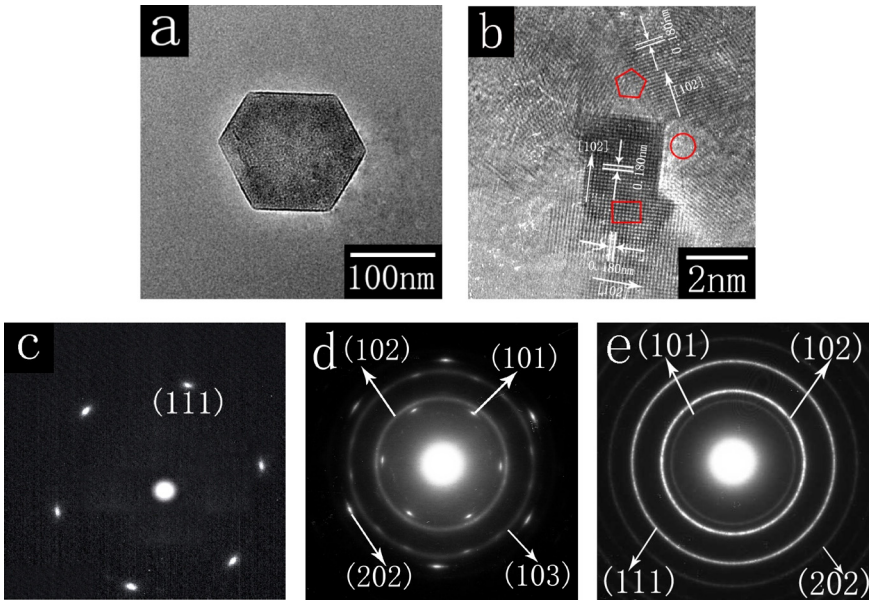

Fig. 3. (a) TEM image of a hexagonal $Mg(OH)_2$ nanoflake. (b) HRTEM of hexagonal $Mg(OH)_2$ nanoflakes. (c)–(e) SAED patterns of hexagonal $Mg(OH)_2$ nanoflakes circled by the rectangle, pentagon and ring in (b), respectively.