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Short communication

Preparation of lamellar magnesium hydroxide nanoparticles via precipitation method

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ABSTRACT

Lamellar magnesium hydroxide (MH) nanoparticles were synthesized via surfactant-free precipitation method using $MgCl_2 \cdot 6H_2O$ and NaOH as raw materials, urea and ethanol as assistant additives. The products were characterized by XRD, TEM, EDS, FTIR and Raman spectroscopy. It is found that the prepared nanoparticles are almost hexagonal lamellas when ethanol is added into the reaction system, which indicates that ethanol is helpful to regulate the morphology of MH nanoparticles. In addition, high-purity MH nanoparticles can be obtained when urea is employed. Both XRD patterns and EDS analysis suggest that the generation of impurities was prevented by adding urea, particularly, under the condition of high concentration raw materials. The possible purification mechanism is due to the coordination interaction of urea with Mg^{2+}, which can prevent the impurity from generating.

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1. Introduction

As one kind of environmentally friendly flame retardant filler, magnesium hydroxide (MH) has attracted great concern in recent years [1–3]. As we know, the shapes of nanoparticles play critical roles in determining their basic properties. For example, MH nanoneedles and nanolamellas are good candidates for functional polymeric composites and fiber hybrid materials as reinforcing agents or flame-retardants [4,5].

During large-scale synthesis, nanoparticles may adopt various shapes [6]. Different shapes of nano-MH have been obtained via different approaches, including precipitation method [7–10], sol-gel method [11], hydrothermal reaction [12–14], and solvothermal modification [15], *etc.* [16–19]. Amongst these methods, the precipitation approaches are more usually employed in practice than others since it is easy-operated and economically acceptable.

In this work, a new route to synthesize large quantity and high-purity lamellar MH nanoparticles is developed. In this approach, ethanol is added into the reaction system for improving the morphology of MH nanoparticles and urea is employed for preventing the generation of impurities [8]. Significantly, there are almost no raw materials wasted in fabrication procedure.

2. Experimental

2.1. Preparation of MH nanoparticles

All chemical agents used in the experiments are of A.R. grade without further purification. 16.0 g NaOH, 1.0 g urea and 8 mL ethanol are dissolved in a three-neck flask containing 50 mL deionized water, and 50 mL $MgCl_2$ solution (4.0 mol·L^{-1}) is then added drop-wise into the flask within 10 min. Afterwards, the mixture solution is kept by mechanical stirring at 60 °C for 1 h and the obtained suspension is consequently aged for 10 h at the same temperature. The precipitates are washed with slightly alkaline water for three times and dried in vacuum at 60 °C. The final products are white crystals and named Sample 1.

The other samples are similarly synthesized. It should be noted that ethanol is unemployed in Samples 2, 3, 4, and urea is not added in Samples 3, 4. In addition, Sample 5 is prepared under the same

Table 1
The main reaction parameters of the samples

	Temperature/ °C	Aging time/h	Urea/g	Ethanol/ml
Sample 1	60	10	1.0	8
Sample 2	60	10	1.0	–
Sample 3	60	10	–	–
Sample 4	60	3	–	–

"–" unemployed.

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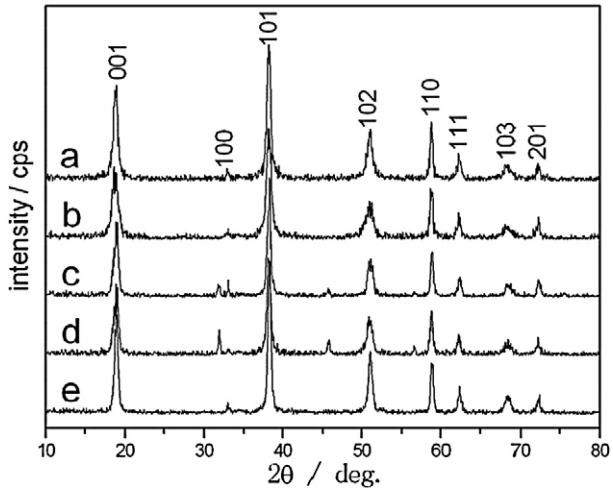

Fig. 1. The XRD patterns of (a) Sample 1, (b) Sample 2, (c) Sample 3, (d) Sample 4 and (e) Sample 5.

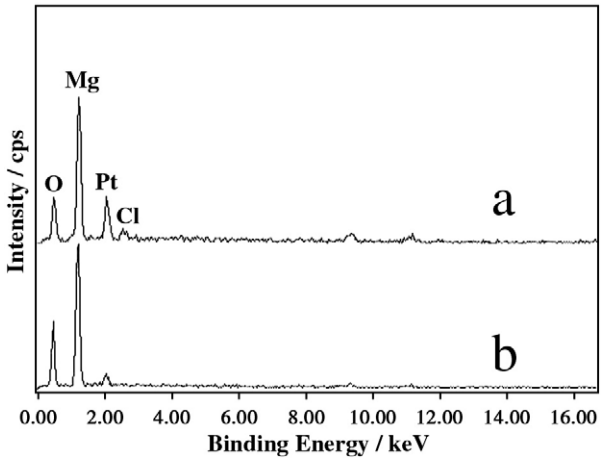

Fig. 3. The EDS analysis of (a) the Sample 1 and (b) the Sample 3.

condition as Sample 4 except that the amounts of $MgCl_2$ and NaOH are decreased by half, respectively. The details are listed in Table 1.

2.2. Characterization

The structures of the as-products were detected by Bruker D8 Advance X-ray diffractometer (XRD) using Cu Kα radiation (λ=0.15406 nm). FTIR spectrum (KBr disc) was recorded on a Bruker Vector 22 spectrometer. Raman spectrum was run on a Renishaw in via laser Raman spectrometer. Energy dispersive X-ray Spectrometer (EDS) analyses were obtained on a JEOL JSM-6380LV Scanning Electron Microscope. TEM images were taken with a JEOL JEM-2100 electron microscope.

3. Results and discussion

The XRD patterns of MH nanoparticles are illustrated in Fig. 1. The diffraction peaks correspond to hexagonal structure MH, according to JCPDS 75-1527. The pattern of Sample 1 (Fig. 1a) indicates a lamellar structure with layers in [0 0 1] direction [8,20], and the thickness of nanoparticles is about 12.5 nm, which is estimated by applying Debye–Scherrer formula, based on full width at half-maximum (FWHM= 0.6370 rad., 2θ=18.75°).

Giorgi *et al.* have reported a similar work on synthesis of MH nanoparticles by precipitation method [9], in which no urea is

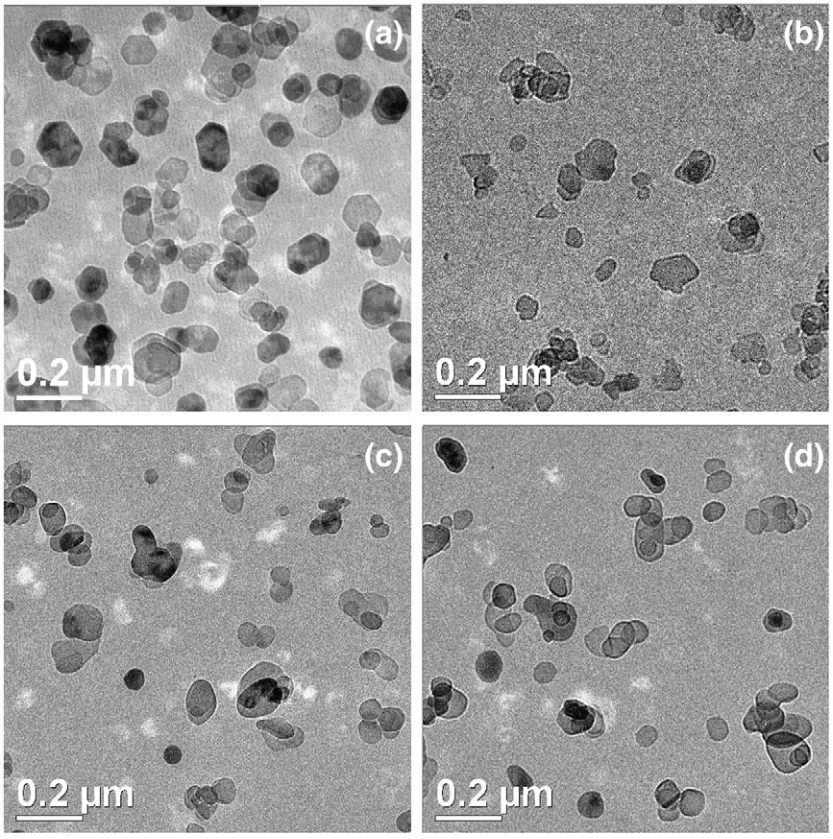

Fig. 2. TEM images of (a) Sample 1, (b) Sample 2, (c) Sample 3 and (d) Sample 4.

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