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A simple and safe method for preparation of $Mg(OH)_2$ nanorods in ambient air

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ABSTRACT

Magnesium hydroxide $Mg(OH)_2$ nanorods with diameters of 90±10nm and lengths of more than 2μms were prepared by a simple route at ~75^oC in ambient air. The approach is based on reaction of magnesium powder and de-ionized water without the use of any harmful, toxic or corrosive reagents. Characterization using X-ray diffraction (XRD) shows single crystalline structure of $Mg(OH)_2$ (brucite) whereas Scanning Electron Microscopy (SEM) showed that nanorods are almost uniform in size. Since only water is used as a solvent as well as a source of oxygen, we believe that this manufacturing process is suitable for mass production of nanostructures safely.

Keywords: *Mg metal; Water; Nanorods; Growth; FESEM*

INTRODUCTION

New paradigms are shrinking our world. Innovations at the intersection of medicine, biotechnology, engineering, physical sciences and information technology are spurring new directions in R&D, commercialization and technology transfer. The future of nanotechnology is likely to continue in this interdisciplinary manner. Nanotechnology is the next industrial revolution and it will radically transform most of all industries in a few years [1]. Magnesium hydroxide $Mg(OH)_2$ is a non toxic, non corrosive, thermally stable and environmental friendly flame retardant which undergoes endothermic dehydration and suppresses fumes in fire conditions with low or zero evolution of toxic or hazardous by products [2]. There has been a growing interest in the use of $Mg(OH)_2$ powders as an additive for the manufacture of flame retardant thermoplastics. It is also used as a neutralizer in the treatment of acidic waste waters and gases that contain sulphuric oxides and also an antacid excipient in pharmaceuticals [3].

The way of assembling the atoms or buildings blocks rationally in one direction is the key point of synthesizing one dimensional nanomaterials. Up to now, a large quantity of nano and microstructures with various exciting shapes have been successfully synthesized. The gas-phase based processes always involve vacuum environment, high temperature and complicated controlling process, which may result in poor dispersion, impurity or the decomposition of the final product and are unfavourable for low-cost and large-scale production. In contrast, the facile solution-based synthesis approaches become a promising choice to address the above problems due to their facile manipulation, low cost, and potential for scale-up by increasing the amount of the reactants. Due to the low growth temperature, the convenience and simplicity in fabrication process, hydrothermal treatment, as a typical solution approach, has been extensively explored for growing metal oxide single crystals with a variety of morphologies, including rods, tubes, disks, flowers, flacks, dumbbells, or other nano- and microstructures by self assembly of nano scaled building blocks [4,5].

$Mg(OH)_2$ nanorods have been prepared by solid arc discharge method [6]. Though this method is mild and convenient but NaCl has been used during the synthesis process which besides having adverse effects on the environment is also toxic. Nanoflowers of $Mg(OH)_2$ have been prepared by hydrothermal method but $MgCl_2$ and $Co(NH_2)_2$ have been used as precursors [7]. The method therefore, is not suitable for mass production keeping in view the initial cost of materials. Plate shaped nano $Mg(OH)_2$ powder have been obtained through calcinations using concentrated sulphuric acids during preparation [8]. An overview of these methods, however, reveals that they involve multistep process and frequent use of amines. Other techniques are technically complex, requires high temperature, expensive experimental set up and/or complicated control process. A simple approach that can avoid organics for large scale production and controlled growth of nanomaterials is, therefore, highly desired.

In our preliminary studies reported earlier, we obtained nanoflakes, nanoflowers of MgO and nanoneedles of $Mg(OH)_2$ on reaction with water by varying reaction parameters like time, temperature and subsequently pressure [9-11]. It was proposed that the growth of nanostructures with different morphologies, sizes, compositions was mainly controlled by temperature and duration of reaction process. Driven by these results, the present studies have been carried out keeping the reaction time and temperature constant. To the best of our knowledge, the technique for the preparation of $Mg(OH)_2$ nanorods without using of any organics reagents at a relatively low temperature of 75^{o}C has not been reported before so far. The synthesis of $Mg(OH)_2$ nanostructures with special morphologies has recently attracted lot of attention because of its huge potential and novel applications.

EXPERIMENTAL

Magnesium powder was used without any preheated producer or any further purification and de-ionized water was prepared in laboratory. 2 mg of magnesium foil was placed in a glass vial containing 20 ml of de-ionized water and the mixture was well sonicated for 15 minutes. The reaction mixture was transferred to glass tube and kept at 75^{o}C in an oven for 12h. After the desired time, the system was allowed to cool down to room temperature. The reaction mixture was then centrifuged to reclaim the precipitated sample and was washed with de-ionized water. After drying in air, the final white powder was characterized using SEM chamber without any further treatment.

Characterization

Phase structure and the purity of the prepared samples was characterized by powder X-ray diffraction (XRD) using a Philips (X'Pert PRO PW-3710) diffractometer with 2θ ranging from 10-80^o, using Cu Kα (λ =0.15141 nm) radiation operated at 40kV and 30mA. The morphology of the products is carried out using Scanning Electron Microscope (SEM, LEO-1530VP), coupled with energy dispersive X-ray spectrometer EDX (Gensis). FTIR spectrum of the samples were determined using Ft-IR 1750 (Perkin Elmer instruments) using palettes of KeBr.

RESULTS AND DISCUSSION

Phase identification

The XRD patterns of the as-prepared samples synthesized at 75°C shown in Figure 1 reveal diffraction peaks of (001), (100), (101), (011), (102), (110) and (103), which are characteristic of the $Mg(OH)_2$. The crystalline phases of $Mg(OH)_2$ are hexagonal with space group P3m1 with the lattice parameters a = 3.1442Å and c = 4.777Å which are close to the reported values corresponding to JCPDS No. 45-0946 are identified unambiguously. The relative broad peaks suggest high crystallinity of the samples. The fact that no discernible peak was identified in the low range of 2θ = 1–10◦ rules out the existence of the amorphous structure. These results are in good agreement with those reported in the literature [12].

Morphology examinations

Figure 2 (a,b) and (c,d) show the low and high magnification SEM images of the prepared samples and confirms that the nanorods were grown at a very high density. The typical diameters of the grown nanorods were in the range of ~90 ± 10nm and the length of few micrometers. It can be seen that the prepared products display the morphology of nanorods with an average diameter of 90nm. Compared with the previous reports the temperature applied in the synthesis is relatively much lower and the synthesis is much cleaner and consistent as can be readily see from micrographs.

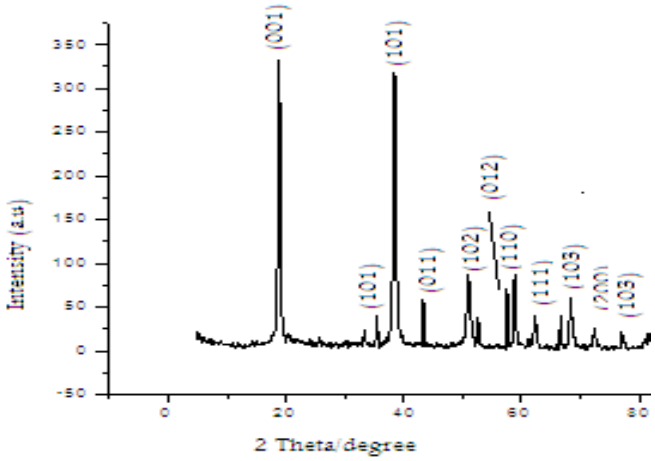

Fig.1. XRD pattern of as prepared sample

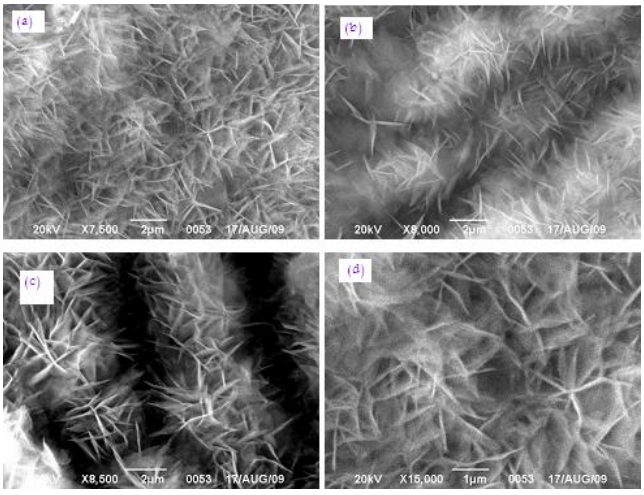

Fig.2. Typical (a,b) low and (c,d) high-resolution SEM images of Mg $(OH)_2$ nanorods.

Composition of sample

The EDX analysis confirmed that the grown nanorods were composed of magnesium and oxygen only as shown in Figure 3. The molecular ratio of Mg:O, calculated from EDX and quantitative analysis data, is close to that of bulk $Mg(OH)_2$ (5% error is attributed to the analysis technique).

The FTIR spectrum

The FTIR spectra of $Mg(OH)_2$ nanorods Figure 4 shows various peaks. The peak at $3350Cm^{-1}$ is due to OH dipole of brucite whereas $996cm^{-1}$ peak indicates the bent vibration of H-O-H. Although the peak at $2850cm^{-1}$ is related to Mg-O, the broad absorption band between 996 and $2850cm^{-1}$ implied the transformation from fee protons into a proton conductive state in brucite.

The formation mechanism

The mechanism for formation of nanostructures can be explained as follows:

$$Mg\ (metal) + H_2O \xrightarrow{\Delta} Mg\ (colloid)$$

$$Mg+2 \xrightarrow{\Delta} Mg(OH)_2 + H_2O$$

Initially at elevated temperatures the concentration of magnesium hydroxide increases correspondingly and magnesium hydroxide nuclei form when the supersaturation level surpasses the critical value. However, the elevated temperatures and pressure are the key factors which kinetically facilitates the decomposition and formation of $Mg(OH)_2$ nuclei. Earlier techniques involving a hydrothermal hydrolysis to produce $Mg(OH)_2$ and subsequent thermal decomposition to obtain MgO nanorods have been well explained. Moreover, water at elevated temperatures plays an essential role in the precursor material transformation because the vapour pressure is much higher and the state of water molecule at elevated temperatures is different from that of room temperature. The solubility and the reactivity of the reactants also change at high pressures and high temperatures and high pressures are favourable for crystallizations. The steam is generated under temperatures to produce a hydrostatic pressure which in turn imposes a profound effect on the ultimate microstructure of the hydroxide thus prepared.

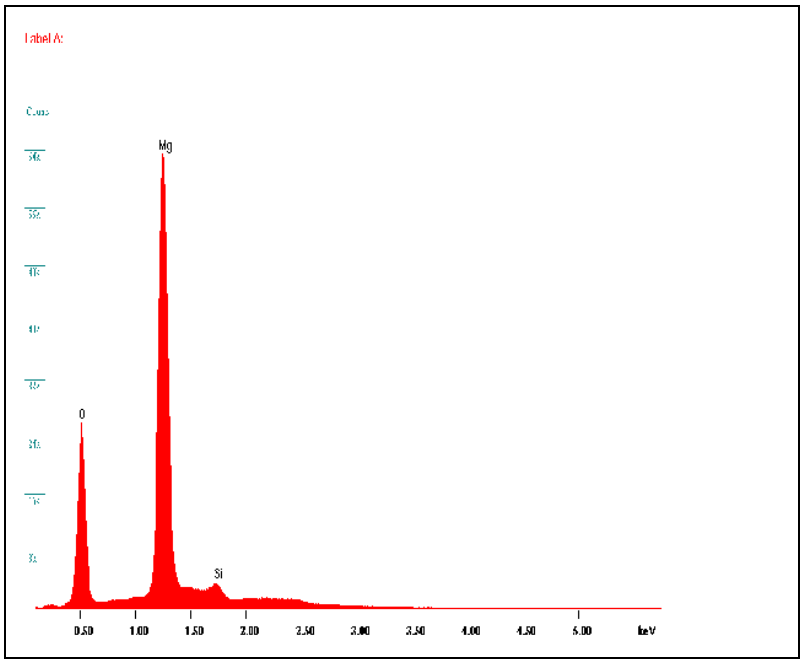

Fig.3. The corresponding EDX analysis confirming the existence of all elements involved in sample

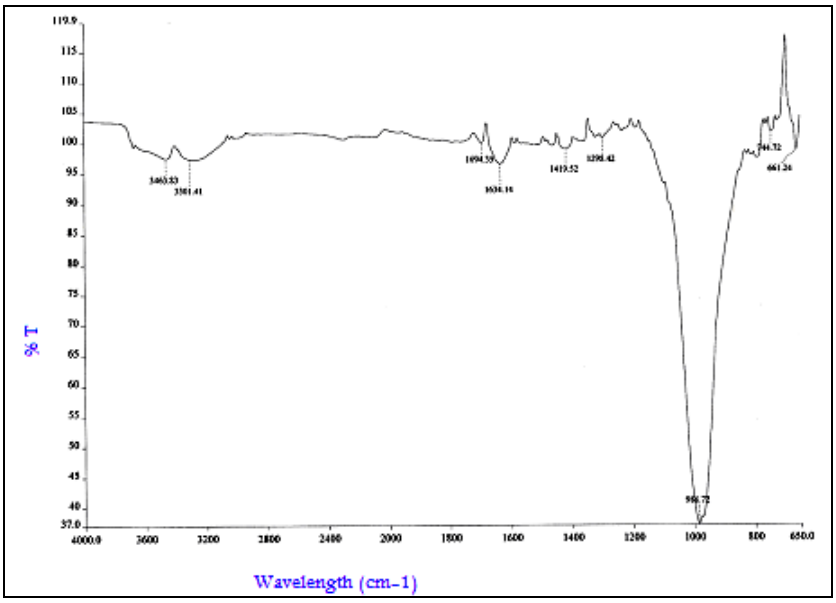

Fig.4. The FTIR spectrum of the samples

CONCLUSION

This study has shown that brucite $Mg(OH)_2$ nanorods can be prepared through simple reaction between Mg powder and de-ionized water at 90^{o}C over a reaction time of 12 hours in atmospheric pressure. This method offers a convenient, safe and efficient route for the preparation of $Mg(OH)_2$ without the use of harmful chemical reagents. The method may be extended to fabricate other metal hydroxide nanomaterials economically.

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