Materials Letters 134 (2014) 210–213

Contents lists available at ScienceDirect

Materials Letters

journal homepage: www.elsevier.com/locate/matlet

Synthesis and characterization of novel organic magnesium salt flame retardant

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ARTICLE INFO

Article history:
Received 3 February 2014
Accepted 12 July 2014
Available online 19 July 2014

Keywords:
Crystal structure
Thermal properties
Magnesium hydroxide
Organic magnesium salt
Reactive flame retardant

ABSTRACT

Magnesium hydroxide (MH), being an environment-friendly flame retardant, are usually added into polymers to obtain satisfying flame retardancy in high loading. Bad compatibility and high loading both cause poor mechanical properties of polymer composites. In this paper, organic groups were introduced into the molecular structure of MH, which can take part in the synthetic reaction of polymer and then be grafted on long chains of polymer. It is promising that the integrated performance of flame retardancy and good mechanic properties can be achieved at the same time. Pure terephthalic acid (PTA) was chosen to react with MH in organic solvent to obtain an organic magnesium salt (OMS) containing organic groups. The obtained OMS was characterized in terms of morphology, particle size, crystal structure, and thermal stability by field-emission scanning electron microscopy (FESEM), X-ray power diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), thermogravimetric analysis (TGA). Moreover, the molecular structure of OMS was verified by mass spectrometry (MS).

1. Introduction

In last decade, there has been a growing interest in the use of magnesium hydroxide powders as environment-friendly flame retardant additives for manufacturing flame retardant thermoplastics, due to its non-toxicity, non-corrosiveness, antacid, thermal stability and being undergoing endothermic dehydration and suppressing fume under fire conditions [1–6]. Being a flame retardant, the following demerits of MH restrict its wide application, such as strong surface polarity, severe agglomeration, poor compatibility with organic substrate and high loading for a desired flame retardant effect, leading to bad mechanical properties of composites. Lots of researchers tried different ways to solve above issues, including ultrafining of particles to increase interfacial area between MH particles and polymer matrix, surface modification to improve the dispersibility and compatibility of MH particles with polymer matrix, and synergism with other flame retardants to enhance flame retardant effect of MH.

However, ultrafine even nanoscaled particles are extremely easy to aggregate to larger ones. So the surface modification is a must to improve the dispersivity and compatibility of ultrafine MH particles. In fact, no matter what treatment has been carried out on MH particles, the physical adding mode has been unchanged. It will be the most challenging thing to add organic groups on the molecular structure of MH, which probably reacts with one or more reactants during the synthesis of polymer. It will be amazing that magnesium hydroxide grafts on long chains of polymer via these organic groups. In this paper, pure terephthalic acid (PTA) was chosen to react with MH in proper solvent at a certain temperature. The obtained product will be reactive flame retardant.

2. Experimental details

All reagents in this work were of analytical grade and used without further purification. Pure terephthalic acid (PTA) and a defined amount of N,N-dimethylformamide (DMF) were added into a 250 ml four-neck flask equipped with a thermometer, a mechanical stirrer, and a refluxing condenser. Then the mixture was heated to a certain temperature under stirring. After PTA being completely dissolved in DMF, MH particles were added in the reactive system (MH:PTA=1:1 mol/mol). Six hours later, the resultant product was vacuum-filtered, and then washed with hot DMF solution. Finally the powdered samples were dried in muffle furnace at 200 °C for 2 h to remove the solvent, named as OMS.

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http://dx.doi.org/10.1016/j.matlet.2014.07.077
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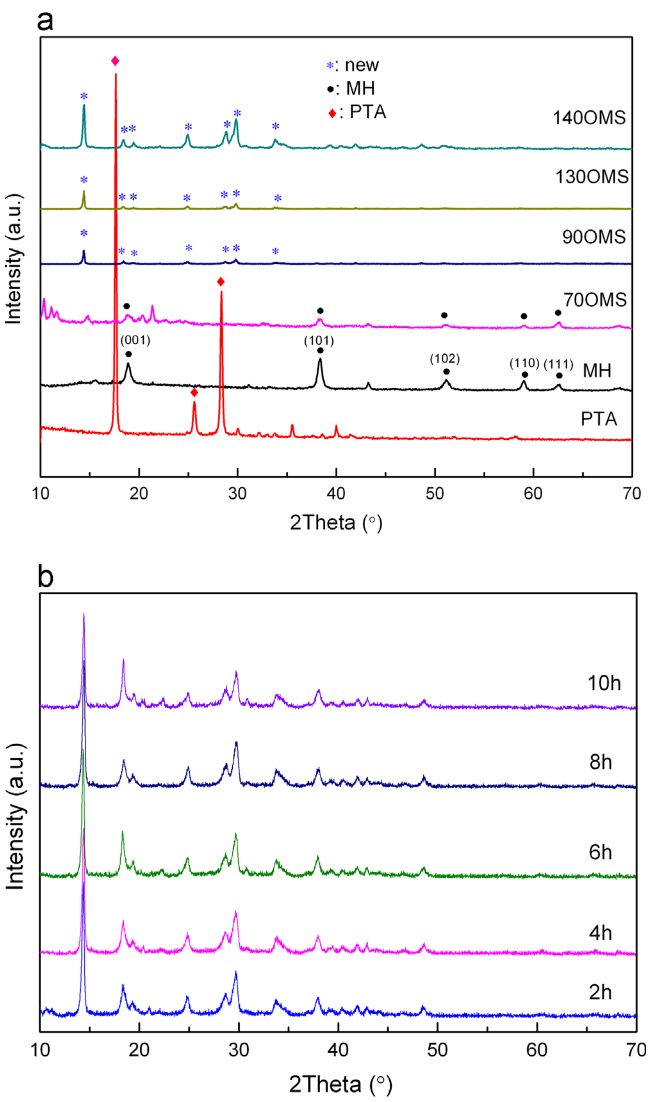

Fig. 1. XRD patterns of OMS obtained at different temperatures for 6 h (a) and at 140 °C for different time (b).

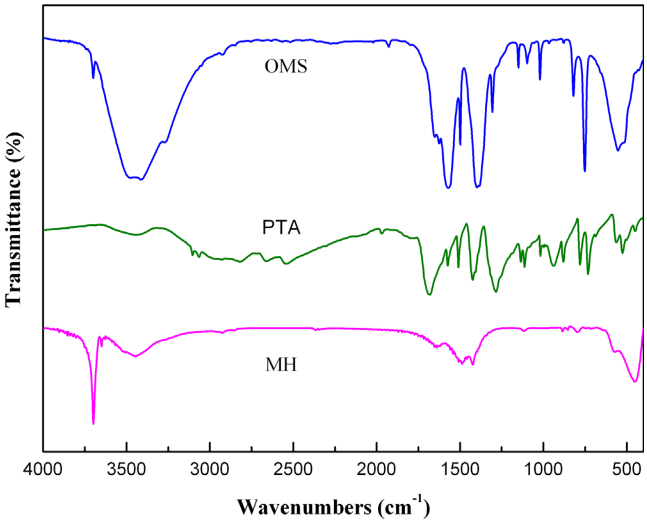

Fig. 2. FT-IR spectra of MH, PTA and OMS.

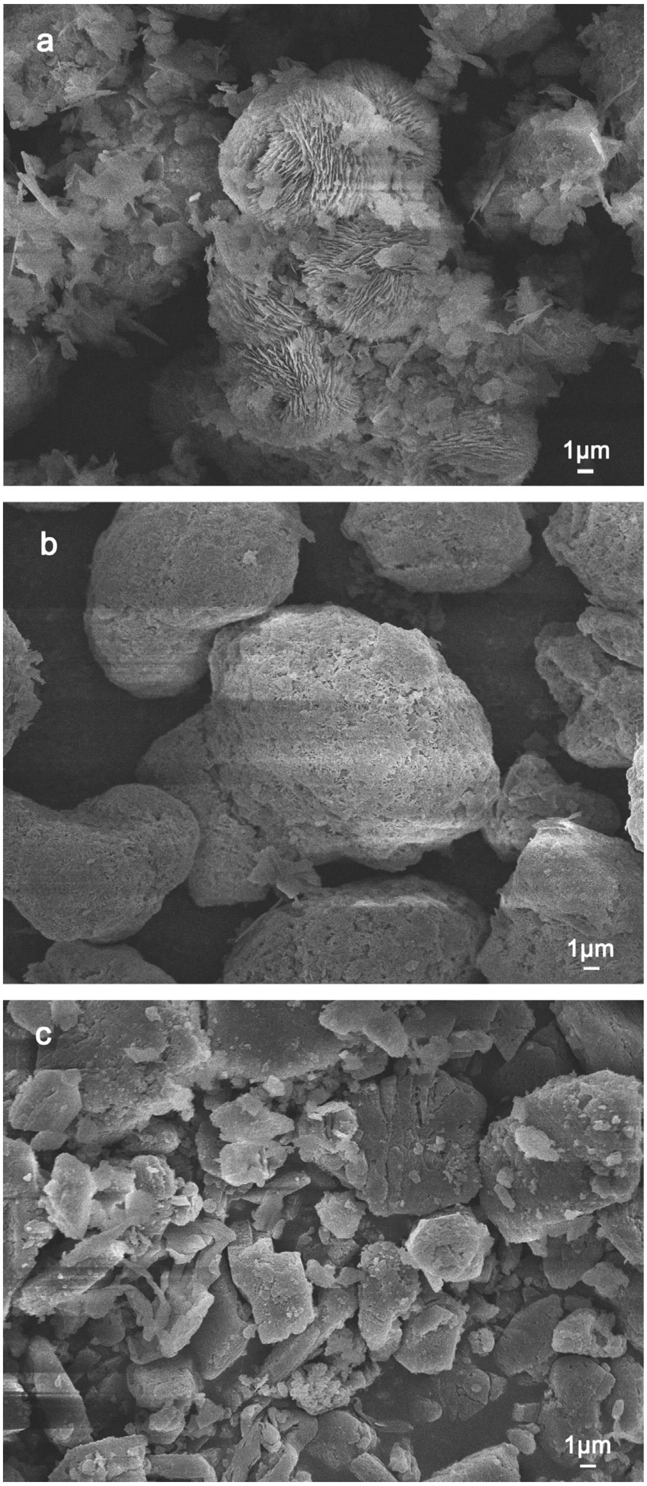

Fig. 3. FESEM images of MH (a), MH heated in DMF (140 °C, 6 h) (b), and OMS (c).

Field-emission scanning electron microscope (FESEM; JSM-6700F, JEOL, Tokyo, Japan) was used to observe the morphologies of different samples. The phase and crystallographic structures of the samples were examined by X-ray diffraction (XRD) on a Y-2000 (Dandong, China) powder diffractometer working with CuKα radiation and Ni filter ($\lambda = 1.54178$ Å) at a scan rate of 0.05 deg/s. FTIR spectrum was recorded on a Bio-Rad (Hercules, CA) FTS-165 spectrometer. Thermogravimetric analysis (TGA) was carried out on a TG-209 thermoanalyzer (Netzsch, Germany), N_2 atmosphere, heating rate 10 °C/min. The mass analyses of different samples were performed on a Varian 320-MS triple quadrupole mass spectrometer.