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Synthesis of zinc hydroxystannate microcapsule for improving flame retardancy and smoke suppression of poly(lactic acid)

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ABSTRACT

In this paper, zinc hydroxystannate (ZHS) was microencapsulated by aluminium phosphate (AlP) to prepare AlP/ZHS microcapsule, it was introduced into polylactic acid (PLA). The flammability of the PLA was characterized by limiting oxygen index (LOI), vertical burning (UL-94), smoke density (SDR) and cone calorimeter test. The results showed that PLA containing 15 wt% AlP/ZHS microcapsule achieved the LOI value of 32.5%, passed the UL-94 V-0 rating, and significantly decreased the peak heat release rate, which imply that the fire safety of PLA has been improved. Meanwhile, the SDR value reached 44%. Thus, AlP/ZHS microcapsule can effectively enhance flame retardancy and smoke suppression, showing potential applications as multifunctional advanced composites.

1. Introduction

Poly(lactic acid) (PLA), one of the most important biodegradable candidate polymers, it is produced by fermentation from renewable resources [1]. At present, as a valuable bio-sourced polymer alternative in long-term applications such as electronics and automobile, PLA is gaining more and more attention [2]. However, this material will be caused fire hazard when the temperature of environment is high enough. Therefore, the electronic industry usually requires the materials with excellent flame retardancy properties.

One way to improve the flame retardancy is to combine polymer matrix with flame retardant fillers. Conventional flame retardants used in polymer are organic halogen compounds, which have been restricted because of the release of toxic substances and a large amount of smoke during combustion [3]. In order to overcome these bottle-necks, flame-retardant PLA has been reported with various nanoparticles, including carbon nanotubes, layered silicates or clays, silica, graphite, etc [4–6]. Ray et al. [7], the effect of montmorillonite on thermal stability and mechanical properties of PLA was discussed. Kim et al. [8], the effect of exfoliated graphite on thermal stability, mechanical and electrical properties of PLA was discussed. Up to now, zinc hydroxystannate (ZHS) has gained much attention because of its non-toxic, environmentally friendly and flame retardancy. However, there are some limits in the manipulation and processing of ZHS such as incompatibility and tending to aggregate, resulting in their nonuniform dispersion and bad compatibility in polymer matrix. To improve this issue, functionalization with coupling agents is a common choice. However, many coupling agents are combustible and lead to the difficulty in the enhancement of flame retardancy and smoke suppression. Hence the development of new highly effective, "green" flame retardants has prompted much attention during the last decade.

In this article, the as proposed method is demonstrated by preparing aluminium phosphate (AlP)/ZHS microcapsule, which was introduced into PLA. It was expected that AlP/ZHS microcapsule can effectively enhance flame retardancy and smoke suppression. It shows great potential for the development of various flame-retardant materials such as PLA.

2. Experimental

2.1. Materials

The phosphoric acid (H_3PO_4), aluminium hydroxide ($Al(OH)_3$), sodium dodecyl sulfate(SDS) and polyoxyethylene octylphenol ether (OP-10) were purchased from Hangzhou Gaojing Fine Chemical Co., Ltd (China). The zinc hydroxystannate (ZHS) were purchased from Hangzhou Baoxin Chemical Co., Ltd (China). The poly(lactic acid) (PLA, 6252D) was obtained from Nature-works Co.,Ltd (USA).

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2.2. Preparation of AlP/ZHS microcapsule

The AlP/ZHS microcapsule was synthesized according to the schematic illustration of the procedure in Fig. 1(1). In detail, SDS (0.01 g), OP-10 (0.1 g), ZHS (5 g) and 100 mL deionized water were added to a 500 mL three-necked flask equipped with a reflux condenser and a thermometer. After ultrasonic vibrations for 10 min, H_3PO_4 (10 g) and $Al(OH)_3$ (10 g) were added to the mixture and the reaction proceeded at 80 C for 1.5 h. Finally, the mixture was removed the solvent and water by spinning evaporation.

2.3. Preparation of PLA composites

In detail, PLA (100 g) and chloroform (50 mL) were mixed at 75 °C for 30 min using mechanical stirring. Then the flame retardant (0%, 5%, 10%, 15%, 20%) was added to the mixture, and then the mixture was further stirred for 1 h at 75 °C. The PLA films were casted on Petri dishes, pilled and dried. These films were about 1 mm thick.

2.4. Characterization

Fourier transform infrared (FTIR) was measured by using Nicolet 5700 spectrometer (Nicolet Instrument Company, USA). The SEM (JSM-5610LV) and TEM (JEM-2100) were used to observe the morphology of samples. Limiting oxygen index (LOI) was measured by using HC-2 oxygen index meter (TESTech Instrument Technologies, China), according to ASTM D2863-2000. Vertical burning rest (UL-94) was measured by using CZF-2 instrument (Jiangning Analysis Instrument Factory, China), according to ASTM D3801. Smoke density was measured by using JCY21 instrument (ESTech Instrument Technologies), according to ASTM D2843-1993. The combustion behavior was measured by using cone calorimeter (Stanton Redcroft CO., Ltd (British)), according to ISO

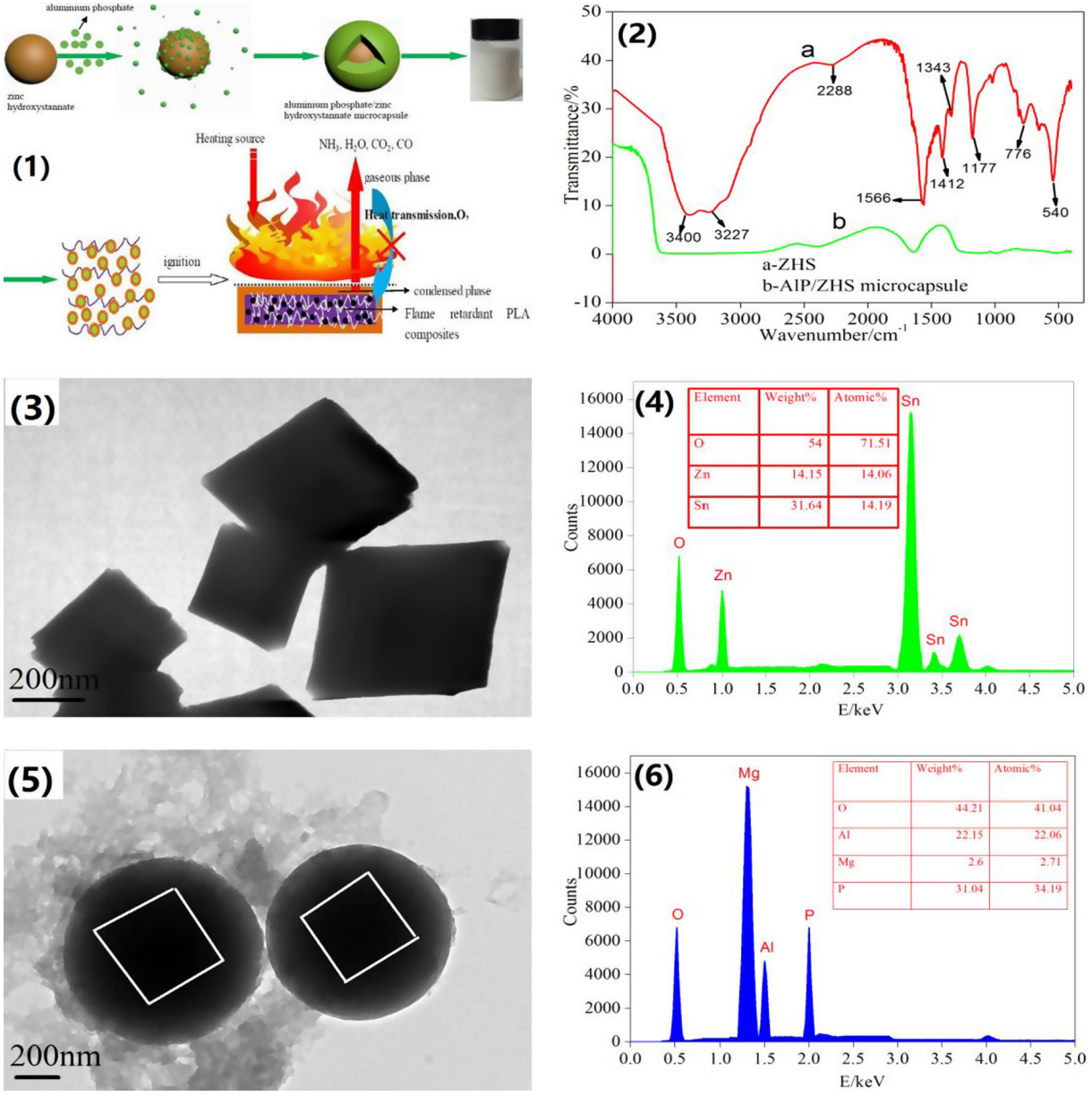

Fig. 1. 1) Synthetic routine of AlP/ZHS microcapsule. 2) FTIR spectra of AlP/ZHS microcapsule. 3) SEM micrographs of ZHS. 4) EDX analysis of ZHS. 5) TEM micrographs of AlP/ZHS microcapsule. 6) EDX analysis of AlP/ZHS microcapsule.