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Exploring the effect of melamine pyrophosphate and aluminum hypophosphite on flame retardant wood flour/polypropylene composites

Panpan Zhao, Chuigen Guo, Liping Li *

College of Materials and Energy, South China Agricultural University, Guangzhou 510642, China

HIGHLIGHTS

- Incorporation of MPP and AHP into the PP/WF composite significantly improved the thermal stability and char forming ability.
- PHRR and THR of the PP/WF composite reduced remarkably by the addition of MPP/AHP.
- The system of MPP/AHP was equipped with perfect effect of gas phase and condensed phase flame retardancy.

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ABSTRACT

In this examine, melamine pyrophosphate (MPP) and aluminum hypophosphite (AHP) were selected as flame retardants. The synergistic effect between the MPP and AHP on flame retardancy and thermal stability of the polypropylene (PP)/wood flour (WF) composite were investigated. The thermal stability and char forming ability were enhanced considerably for the PP/WF composite with 20 wt% MPP/AHP (3:1), which were indicated by the thermal gravimetric analysis (TG), along with reductions in the heat release rate (HRR) and the smoke production release rate (SPR). The outcomes of TG, scanning electron microscopy and energy-dispersive X-ray spectroscopy revealed that there were inert gases as well as more phosphates and phosphoric acid formed. Which effectively diluted flammable gases and enabled more intumescent char residue to form. In summary, the system of MPP/AHP was equipped with perfect effect of gas phase and condensed phase flame retardancy, endowing PP/WF composite with outstanding capacity of inflaming retarding.

1. Introduction

Nowadays, wood-plastic composites (WPCs) as potential material had captured both researchers' and manufacturers' attention with excellent characteristics, including recyclability, favorable machinability, low cost, dimensional stability, high stiffness and strength [1–3]. Due to those excellent performances, it had applied to various fields such as furniture, decking, building industries and automobile products etc. [4–6]. However, we cannot ignore the serious limitation caused by high fire risk, which resulted from the two main flammable compositions (natural fibers and thermoplastic plastics) [7–8]. Therefore, it was vital to impart flame retardancy of WPCs, and many efforts had been endeavored in it [9–12].

Intumescent flame retardants (IFRs) have aroused great attention in flame retarded WPCs in recent years, owing to their more environmental-friendly, anti-dripping and lower smoke compared with the traditional halogen-containing flame retardants [13–15]. Typically, ammonium polyphosphate (APP) was the most effective and widely used IFRs for WPCs, which possessed both acid source and blowing agent, and wood flour (WF) as a charring agent [16–19]. However, the significant disadvantage was that APP can deteriorate the mechanical properties of WPCs due to its hygroscopicity and poor compatibility with organic matrix [2]. One solution to the problem was to employ a new FR possessing acid source and blowing agent such as melamine pyrophosphate (MPP), which had excellent thermostability and water-resistance characteristics widely used to flame retardant nylon [20–22]. However, little work had been done for MPP flame retarded WPCs. Yoshihiko et al. [5] had examined how flame retardants such as ammonium polyphosphate (APP), melamine polyphosphate (MPP) and aluminum hydroxide to impact the flame retardance and mechanical properties of WPCs. It was shared that WPCs with MPP showed the better mechanical properties, but lower flame retardancy than WPCs with APP. Therefore, it was an effective way that seeking a synergistic agent of MPP to overcome this problem. Aluminum hypophosphite

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* Corresponding author.
E-mail address: lilipingguo@126.com (L. Li).

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(AHP) has attracted great attention as an efficient environmental friendly and halogen-free flame retardant with the high content of phosphorus [23–25], which may be a good synergistic agent of MPP.

The synergistic effect between MPP and AHP on the PP/WF composite were investigated by limiting oxygen index (LOI), cone calorimetry tests and thermogravimetry analysis (TG). To further study the synergistic mechanism between MPP and AHP, the formation of char layers and their microstructure of WPCs after burning were studied by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDX).

2. Experiment

2.1. Materials

Polypropylene (PP) was purchased from Daqing Petro Chemical Company in China with the density of 0.89–0.91 g/cm^3 and the melt flow rate of 8 g/10 min (230 °C). Wood flour (WF) was purchased from Harbin Yongxu in China with 80 mesh sieving. Antioxidants–1010 was purchased from Jiangsu Hangguang Company in China. Polypropylene grafted with maleic anhydride (MA-g-PP) was purchased from Hangzhou Haiyi polymer material Ltd. in China. Melamine polyphosphate (MPP) was purchased from Shenzhen Crystal Chemical Co. in China. Aluminum hypophosphite (AHP) was purchased from Qingdao Fu Muslim Chemical Technology Co. in China.

2.2. Sample preparation

The WF was dried at 105 °C for 5–8 h in an electric thermostatic drying oven (DHG-9075A, produced by Shanghai Yiheng Instrument co., Ltd.). PP, AHP and MPP were dried at 60 °C for 4–6 h. Then the dried materials were mixed uniformly in a high speed grinder (FW-80, produced by Tianjin Teste Instrument Co., Ltd.). The samples were prepared by melt blending in the torque rheometer (RM-200, produced by Harbaugh Electric Manufacturing Co., Ltd.) at 180 °C for 8 min, and pressed on a curing machine at 180 °C for 20 s, and then the sheets were formed by high-pressure cooling to test. Table 1 showed the formula of samples. In order to explore the effect of flame retardants on PP/WF composites, the proportion of PP and WF were settled as 2:3.

2.3. Measurements

2.3.1. The limited oxygen index (LOI)

LOI was measured by a JF-3 Oxygen Index Meter (Jiangning Analysis Instrument Company, China) as standard ASTM D-2863. The specimen dimension used was 130 × 6.5 × 3 mm^3.

2.3.2. The UL-94 vertical tests

The UL-94 vertical test was measured by a CFZ-3 Vertical Burning Instrument (Jiangning Analysis Instrument Company, China) as standard ASTM D-3801. The dimensions of samples tested were 1 25 × 13 × 3.2 mm^3.

2.3.3. Thermogravimetric analysis (TG)

TG was measured using a Pyris 1 Thermal Analyzer (Perkin–Elmer Company, US). Samples were measured under nitrogen at a heating rate of 10 °C/min with a mass of 3–5 mg from 30 °C to 800 °C.

2.3.4. Scanning electron microscopy (SEM)

The morphologies of char residue were observed using a Quanta200 Scanning Electron Microscopy (FEI Company, England). The thickness of specimens was about 2–3 mm, fixed on the sample stage and coated with a conductive layer of gold at the acceleration voltage of 5 kV.

2.3.5. Energy-dispersive X-ray spectroscopy (EDX)

The element content of surface residue was examined by INCA type EDX instrument (the British Oxford Instrument Company). Specimens were coated with a conductive layer of gold.

2.3.6. Cone calorimeter

The cone calorimeter (CONE) tests were executed according to the ASTME 1354-2009 standard. The specimen was wrapped in aluminum foil and placed horizontally with external heat flux of 50 kW·m^{-2}. The dimension of specimen used was 100 × 100 × 3 mm^3.

3. Results and discussion

3.1. LOI and UL-94 vertical tests

The LOI values and UL-94 rating for the samples were summarized in Table 2. According to the results, the sample 1 was flammable in nature with a low LOI value of 23.7 vol%, no UL-94 rating and serious flaming dripping. The LOI values of sample 2 and sample 3 were 27.4 and 26.3 vol% respectively, but they had no UL-94 rating as well.

This indicated that MPP and AHP had little effect on flame retardant PP/WF composite when adding individually. When MPP and AHP were imported simultaneously, the LOI values were higher than that of sample 2 and sample 3. Especially, for sample 6, when the mass ratio of MPP and AHP was 3:1, the LOI value was enhanced by 23.2 vol% with no flaming dripping, and the UL-94 test passed V-0 rating. According to LOI and UL-94 tests, the dual-flame retardant systems showed better flame retardance compared with single-flame retardant systems. Hence, the MPP/AHP (3:1) system demonstrated high efficiency and synergistic effect on PP/WF composite.

Table 1
Components of PP/WF composites.

Samples	PP/wt%	WF/wt%	MA-g-PP/wt%	Antioxidant 1010/wt%	MPP/wt%	AHP/wt%	Ratio (MPP:AHP)
1	36.0	54.0	9.0	1.0	0	0	–
2	28.0	42.0	9.0	1.0	20.0	0	–
3	28.0	42.0	9.0	1.0	0	20.0	–
4	28.0	42.0	9.0	1.0	17.5	2.5	7:1
5	28.0	42.0	9.0	1.0	16.7	3.3	5:1
6	28.0	42.0	9.0	1.0	15.0	5.0	3:1
7	28.0	42.0	9.0	1.0	10.0	10.0	1:1
8	28.0	42.0	9.0	1.0	5.0	15.0	1:3
9	28.0	42.0	9.0	1.0	3.3	16.7	1:5
10	28.0	42.0	9.0	1.0	2.5	17.5	1:7