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Synergistic effect of intumescent flame retardant and expandable graphite on mechanical and flame-retardant properties of wood flour-polypropylene composites

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HIGHLIGHTS

- The flame retardancy of the WPC were improved greatly when CFA was introduced.
- The mechanical properties of WPC were improved greatly by adding m-TMI-g-PP.
- The synergetic systems could reduce the HRR, SPR and mass loss of WPC.
- The special microstructure was observed after burning of the expansion system by SEM.

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ABSTRACT

In this article, expandable graphite (EG) and intumescent flame retardant (IFR) that consist of a novel triazines char forming agent (CFA) and ammonium polyphosphate (APP) was chosen in wood flour-polypropylene composites. The synergistic effect between the EG and IFR on mechanical properties, flame retardancy, and thermal degradation of WPC were investigated. The WPC with 25 wt% of EG/IFR (2:3) had the highest strength in the flexural, izod impact, and thermal stability. The ratio showed modest improvements in the Flame retardant performance as indicated by reductions in the heat release rate and the smoke production release rate. SEM results showed that a compact and thick char layer was formed in the ratio, which hindered the transfer of heat flow and combustible gases in the condensed phase. Thus, the combination of EG and IFR had been proved to be a promising flame retardant system for WPC.

1. Introduction

Wood–plastic composites (WPC) are made of wood fiber/flour and thermoplastic(s) (includes PE, PP, PVC, etc.). Currently several commercial WPC are manufactured for the residential construction industry, primarily as lumber for decking and railing systems [1]. Bringing those two materials together moderates their weaknesses. The positive growth in WPC decking has led manufacturers to introduce residential construction applications. Further expansion into the residential construction industry and development of applications for the furniture industry require an understanding of the fire performance of the WPC [2]. The types of plastics normally used in WPC formulations have higher fire hazard properties than wood alone, as plastic has a higher chemical heat content and can melt. Improving of fire retardancy of the composite has been become important in order to comply with the safety requirements of the WPC products [3]. Polymers employed in WPC, burn and drip in case of fire leading to a very risky scenario. Thus, some people are becoming increasingly concerned with the fire performance of WPC [4]. Stark et al. [5] evaluated the effect of five additive-type fire retardants on fire performance and found that magnesium hydroxide and ammonium polyphosphate improved the fire performance of WPC the most while a bromine-based fire retardant and zinc borate improved fire performance the least. Seefeldt et al. [6] found that a new flame retardant acts similar to APP, but as regards the amount of phosphorus inside the material, FR showed even better results than APP. Li and He [7] investigated the flame retardancy and thermal degradation of LLDPE-Wood fiber composites, APP and melamine phosphate (MP) or pentaerythritol (PER) were used as FR, and experimental results demonstrated that APP is an effective FR for LLDPE-wood fiber composites by promoting char formation of the composites.

On recent years, there have been increasing concerns about toxic gases and smoking evolved during the burning of halogenated flame retardant materials. Legislation has limited their uses

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for environmental and safety reasons, which has led to useful worldwide of halogen-free flame retardant materials [8]. The intumescent flame retardant (IFR) systems have attracted great attention because they are halogen-free, environmental friendly and highly effective. An IFR system is usually composed of three elements, an acid source, a blowing agent and a carbonic source. The intumescent char produced from the synergistic interaction among these elements protected underlying materials from heat and oxygen [9]. Upon heating, these three active ingredients form a multicellular swollen char layer, which slows down the heat and mass transfers to interrupt the degradation of a polymer. The mixture of ammonium polyphosphate (APP), pentaerythritol (PER), and melamine is a typical IFR system [10], which has been successfully used in fire-retardant intumescent coatings, due to its low smoke and toxic gases production during burning. Nevertheless, these blowing agents and carbonic sources have drawbacks such as moisture absorption, exudation, and incompatibility with the polymeric matrix [11]. At present, a novel triazine polymer which synthesized was used as both a charring agent and a foaming agent in IFR, named as a novel charring–foaming agent (CFA) [12]. The CFA shows a good ability of char formation itself. The research demonstrated that the IFR consisting of CFA and APP is very effective in flame retardancy of PP.

To a certain extent, the IFR (APP/CFA) has overcome some deficiencies of the traditional IFR system. However, low efficiency, high loading and high cost still exist, so it is imperative to develop new efficient IFR system for WPC. As a new generation of intumescent additives, expandable graphite (EG) with high flame retardant efficiency and low cost is used in a growing number of fire retardant applications as a blowing agent and carbonization compound [13,14]. EG is an intercalated graphite compound in which some oxidants like sulfuric acid and potassium permanganate are inserted between the carbon layers of graphite. When exposed to heat, EG expands and generates voluminous insulating layers, thus improving fire-retardant effect of the polymeric matrix [15,16].

Therefore, EG is widely used as synergist to increase significant char yield and promote the thermal stability of the barrier of IFR and reduce the cost of IFR due to its abundant source, simple preparation, and low cost in many polymers [17]. Recently, literature about EG as a flame retardant in polymers have been published, whereas the study of EG and its interaction with IFR in PP is still under investigation [18,19].

In this study, a new IFR (APP/CFA = 4:1) and EG was chosen which was used on improving combustion properties of WPC. The mechanical properties, flame retardancy, and thermal decomposition were studied based on mechanical instrument, oxygen index meter, vertical burning tester, cone calorimetry, and thermo-gravimetric analysis (TGA). The surface morphology of char residues of the flame-retardant WPC after cone calorimetry test was examined by SEM.

Table 1
Components of samples.

Samples	PP/g	WF/g	*m*-TMI-g-PP/g	1010/g	EG/g	APP/g	CFA/g	Ratio (EG/IFR)
1	40	60	9	1	0	0	0	–
2	40	60	9	1	25	0	0	–
3	40	60	9	1	20	4	1	4:1
4	40	60	9	1	15	8	2	3:2
5	40	60	9	1	10	12	3	2:3
6	40	60	9	1	5	16	4	1:4
7	40	60	9	1	0	20	5	–
8	40	60	9	1	0	25	0	–
9	40	60	9	1	0	0	25	–

2. Experiment

2.1. Materials

Wood flour (WF) was supplied by Harbin Yongxu (Harbin, China). Polypropylene (PP) was supplied by Daqing PetroChemical Company (Daqing, China). APP was supplied by Hengyu (Tianjin, China). Antioxidant-1010 was supplied by Jiangsu Hanguang (Jiangsu, China). Polypropylene grafted with *m*-isopropenyl-α,α-dimethylbenzyl-isocyanate (*m*-TMI-g-PP) and the CFA were prepared in our laboratory. EG (particle size: 300 μm) with a purity of >99% were provided from Yichang Xincheng Graphite, Co., Ltd., China.

2.2. The preparation of samples

The WF, APP and CFA were dried at 105 °C for 8 h in an oven before the experiment. A fixed ratio of amount of PP/WF/*m*-TMI-g-PP and PP/WF/*m*-TMI-g-PP (40:60:9) was used through the treatment. The IFR (APP/CFA) was used as a flame-retardant component in WPC. The components of samples were shown in Table 1. The samples were prepared on a SK-100 two-roll mill (produced by Harbin Special Plastic, China) at 175 °C for 20 min, then pressed on a curing machine at 175°C for 1 min to form sheets for testing.

2.3. Mechanical properties test

The tensile strength, flexural strength was performed using a TA-20 computer controlled universal testing machine (produced by Shenzhen Reger Instrument, China). The Izod impact strength was carried on a XJC-25D impact testing machine (produced by Chengde Precision Testing Machine, China). Five specimens were tested to obtain the average value for each treatment. Tensile strength tests were examined according to ASTM D638 with a crosshead speed of 5 mm min^{-1}. Flexural strength tests were performed according to ASTM D790 with a crosshead speed of 2 mm min^{-1} and a support span length of 64 mm. Izod impact strength tests were conducted according to ASTM D256.

2.4. Flame retardancy test

The Limited Oxygen Index (LOI) was measured by a JF-3 oxygen index meter (Jiangning Analysis Instrument Company, China). The LOI test was performed according to the ASTM D 2836.

Table 2
Mechanical properties of WF/PP composites.

Samples	Tensile strength (MPa)	Elastic modulus (MPa)	Elongation at break (%)	Flexural strength (MPa)	Flexural modulus (MPa)	Notched impact strength (kJ m^{-2})
1	26.2	249.2	17.9	60.2	3627.3	2.3
2	21.3	177.3	15.6	51.0	2675.3	1.9
3	22.6	190.9	15.6	54.5	3376.5	2.3
4	23.9	218.4	15.7	57.2	3588.8	2.4
5	25.7	238.5	16.8	65.8	3892.9	2.7
6	23.2	204.5	16.0	58.1	3617.1	2.6
7	23.0	195.2	15.5	52.5	2788.2	2.2
8	23.8	215.3	15.8	56.8	3498.1	2.0
9	24.6	221.0	16.6	54.3	3101.6	1.9