

Effect of Different Metallic Hydroxides on Flame-Retardant Properties of Low Density Polyethylene/Melamine Polyphosphate/Starch Composites

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ABSTRACT: The flame retardancy of synthesized melamine polyphosphate (MPP) in combination with starch (ST) and different metallic hydroxides was investigated in low density polyethylene (LDPE) by limiting oxygen index (LOI) and vertical burning test. The results indicated that the LOI value of composite comprising $\text{Al}(\text{OH})_3$ (ATH) was higher than those of composites at the same additive loading with $\text{Mg}(\text{OH})_2$ (MH) or $\text{Fe}(\text{OH})_3$ (FH), which increased from 22 to 27%. And the composite comprising ATH passed V1 rating without causing molten drops. In addition, thermostability and morphology were characterized by differential scanning calorimeter (DSC), thermogravimetry (TG), derivative thermogravimetry (DTG), and scanning electron microscope (SEM). The results demon-

strated that the crystallization of the composites remained unaffected after the incorporation of metallic hydroxide. The thermal degradation behavior of LDPE composites and the morphology of residual charred layer were changed. It also can be concluded that there was a synergy between certain metallic hydroxide and MPP after analyzing the residual charred layer using X-ray diffraction (XRD). © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 122: 3263–3269, 2011

Key words: melamine polyphosphate; metal hydroxide; flame retardance; low density polyethylene; thermal properties

INTRODUCTION

Polyethylene (PE) has been used as one of general-purpose plastics due to its chemical resistance, good mechanical properties, excellent electrical insulation and absence of toxicity, and so on. But combustibility and melt dripping limit its use in automobile, electron component and some other fields. How to improve the poor flame resistance of PE has become a constant challenge.

Recently, Chen and Wang¹ reviewed the development of flame retardant in china and introduced some kinds of flame retardants. To achieve good flame resistance, the addition of flame retardants was one of effective measures. Natural mineral (like vermiculite, perlite, and Kaolinite), metallic hydroxide, ammonium phosphate and its derivatives are commonly used as flame retardant due to their low cost. The vermiculite/perlite cement based mortar could provide fire protection to glass fiber reinforced polymer pultruded profiles, which are used as building materials.² $\text{Al}(\text{OH})_3$ (ATH) and

$\text{Mg}(\text{OH})_2$ (MH) are the most common flame retardants. It has been reported that zinc borate and zinc oxide have synergistic effect on polypropylene (PP)/ATH/MH systems and polyvinyl chloride (PVC) treated with ATH, respectively.^{3,4} And through the study of thermal behavior and flammability, it was found that the content of zinc oxide influences the flame retardancy of PVC treated with ATH directly.⁴ Furthermore, different MH also influences the thermal stability and thermal oxidative degradation kinetics in PP composites.⁵ Some halogen containing compounds, usually in conjunction with antimony trioxide, are also used as flame retardant. For example, decabromodiphenyl ether in combination with antimony trioxide has been reported having better effect on controlled pyrolysis of high-impact polystyrene mixed with polyolefin.⁶

However, inorganic flame retardants influence the mechanical properties of polymer resin because of the loading amount and low efficiency. What's more, the halogenated flame retardants produce a lot of poisonous gases, which increased the risk of fire. Therefore, it is needed to find a new style flame retardant, which could overcome all those inadequacies and possess operational retardant effectiveness. Intumescent flame retardant (IFR), as one of halogen-free flame retardant systems with low smoke

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TABLE I
Formulations and Flame Retardancy Test Results of Flame-Retarded Composites

Sample	LDPE (wt %)	MPP (wt %)	ST (wt %)	MH (wt %)	ATH (wt %)	FH (wt %)	LOI (%)	UL-94 test	
								Dripping	Rating
0	100	0	0	0	0	0	17.0	Yes	No rating
1	85	5	10	0	0	0	19.5	Yes	No rating
2	80	10	10	0	0	0	20.5	Yes	No rating
3	75	15	10	0	0	0	20.5	Yes	No rating
4	70	20	10	0	0	0	22.0	Yes	No rating
5	65	25	10	0	0	0	22.0	Yes	No rating
6	65	20	10	5	0	0	24.0	Yes	No rating
7	65	20	10	0	5	0	23.5	Yes	No rating
8	65	20	10	0	0	5	23.5	Yes	No rating
9	60	20	10	10	0	0	25.5	Yes	V-2
10	60	20	10	0	10	0	27.0	No	V-1
11	60	20	10	0	0	10	25.0	No	V-2

density coefficient and limited poisonous gas emission can overcome the inadequacies of traditional flame retardants within a certain range, which normally contains three components, i.e., an acid source, a carbonization agent, and a blowing agent.^{7,8} Previous investigation showed that PP comprising melamine phosphate (MP) and pentaerythritol (PER), dipentaerythritol (DPER) or tripenaerythritol (TPER) could pass vertical burning test and possess certain flame retardancy.⁸ The flame retardancy of melamine polyphosphate (MPP) flame retarded polyamide6 (PA6)/inorganic siliciferous is related to the different geometrical form of inorganic siliciferous.⁹ The synergistic effect between a derivative of triazines and ammonium polyphosphate on the flame retardant and antidripping properties of polylactide has also been verified.¹⁰ Some intumescent flame retardant systems consisting of ammonium polyphosphate (APP), MP, PER or other materials were used in low density polyethylene (LDPE) composites. Those IFR systems can obviously improve the thermostability of composites and the compatibility of flame retardants.^{11–13}

Melamine polyphosphate (MPP) is a derivative of MP and widely used as the flame retardant of polyamide (PA). It has a higher decomposition temperature and much less water solubility compared with MP and APP. The main problems associated with MPP was its poor compatibility with polymer resin. It has been reported that MPP can be used as the flame retardant of PP, PE and PA.^{7,14,15} But there are few investigations on MPP as a flame retardant in combination with metallic hydroxides.

In this study, the objectives were to measure the flame retardant activity of MPP through analyzing the flammability of composites comprising different contents of MPP and to investigate the flame retardant properties of composites comprising different metallic hydroxides. The flame retardancy, mechanical properties, thermal behavior and morphological

structure were studied to verify the flame retardant activity of MPP and its synergy with certain metallic hydroxide in this flame retardant system.

EXPERIMENTAL

Materials

Low density polyethylene (2426H) was purchased from Sinopec Group guangdong Maoming Petrochemical, China. Melamine phosphate (MP) was supplied by Jinghui Chemical Institute, China. Starch was purchased from Foshan Gaohao Starch Factory, China. MH, ATH, and FH were provided by Sinopharm Chemical Reagent, China.

Synthesis of MPP

To avoid the interference of water, MP was firstly dried in a drying oven at 95°C for 10 h. The dried MP was put into a muffle furnace, held at 320°C for 8 h and then cooled down to room temperature. After fully ground, MPP was obtained.

Preparation of LDPE composites

All original materials were blended in a high-speed mixer. The blends of LDPE with various amount of MPP, ST, or metallic hydroxides were prepared using a twin-screw extruder (SHJ-20) at a temperature range of 140–150°C. Samples for testing were prepared using a plastics injection machine (TY-200). The formulations and flame retardancy testing results of composites were given in Table I.

Characterization

The Fourier transform infrared (FT-IR) spectrum of MPP was obtained using a VERTEX 70 FT-IR-spectrometer (BRUKER OPTICS, Germany).

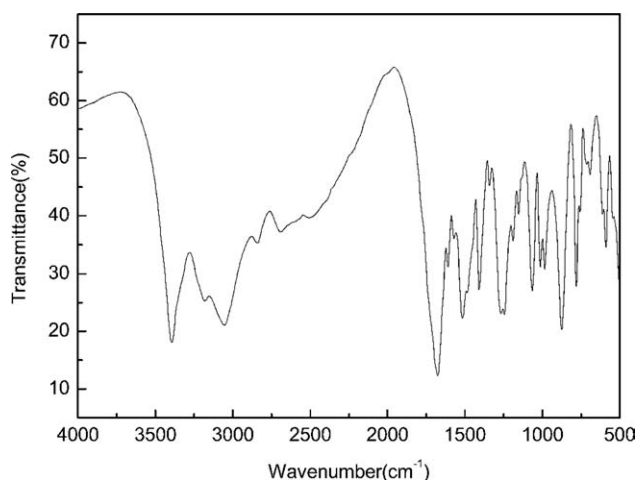


Figure 1 FTIR spectrum of synthesized MPP.

The mechanical properties including tensile strength and flexural strength were measured using a CMT-4104 instrument (MTS, China) at room temperature. Izod impact strength was measured using an XJU-22 izod impact testing machine (Xinma Test Instrument, China). All tests followed corresponding standards.

The LOI values were measured on a HC-2C oxygen index meter (Jiangning Analysis instrument company, China) according to ASTM D2863. Vertical burning tests were conducted on a CZF-2 vertical burning test instrument (Jiangning Analysis instrument company, China) according to the UL94 test standard.

The thermogravimetry (TG), derivative thermogravimetry (DTG) and differential scanning calorimetry (DSC) were performed on a Netzsch STA 449C (Netzsch, Germany) with a heating rate of 10°C/min and a dynamic nitrogen flow of 10 mL/min. DSC was performed with a temperature ranging from 50 to 200°C. TG and DTG were performed with a temperature ranging from 50 to 700°C.

The surface of residual charred layer which was from the burned specimen in UL 94 test were coated with a conductive gold layer and observed by a JEOL JSM-5610LV scanning electron microscope (SEM).

X-ray diffraction (XRD) was performed on an X-ray diffractometer (X'Pert PRO DY2198) with Cu K α radiation to study the composition of the residual charred layer.

RESULTS AND DISCUSSION

FTIR analysis of synthesized MPP

Figure 1 was the FT-IR spectrum of the synthesized MPP. The characteristic peaks of MPP were observed at 3181 and 2840 cm⁻¹ assigned to the

vibration absorption of NH³⁺. The peaks observed at 2690 and 985 cm⁻¹ were assigned to the vibration absorption of O—H and P—O groups in P—O—H group, 1409 cm⁻¹ assigned to the vibration of C—N groups of the triazine rings like melamine, 1268 and 1190 cm⁻¹ assigned to the vibration of P=O bonds, and 876 cm⁻¹ assigned to the vibration of P—O—P bonds. It can be also seen the characteristic absorption of melamine pyrophosphate at 3394, 1572, and 1342 cm⁻¹.^{15,16} The analysis showed that the synthesized MPP was composed of melamine pyrophosphate. The main reason associated with this phenomenon concerns the reaction process: intermolecular dehydration process led to a transition, i.e., MP becoming melamine pyrophosphate firstly during the thermal polymerization at a certain temperature. Then the intermediates were dehydrated to become ultimate production.

Mechanical property and flammability analysis

To measure the flame retardant activity of MPP, composites (LDPE/MPP/ST) comprising different contents of MPP (5, 10, 15, 20, and 25 wt %) were prepared. It is well known that the filler can affect the mechanical property of resin matrix. In this work the combination of mechanical property (CMP) was defined to analyze the mechanical property of composites, which can be expressed by the following equation:

$$CMP = T/T_{Ldpe} + F/F_{Ldpe} + I/I_{Ldpe}$$

where T , F , and I are tensile strength, flexural strength, and impact strength, respectively. So the CMP of LDPE matrix is 3. The relationship between CMP and content of MPP was presented in Figure 2. It can be seen that with the increase of MPP content, CMP increased variously. CMP of composite

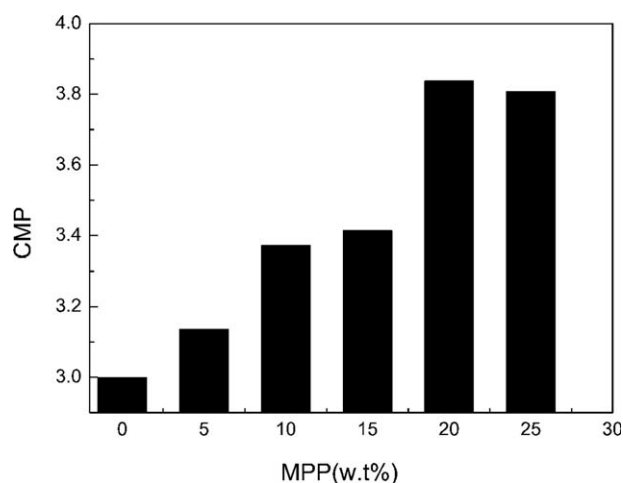


Figure 2 Relationship between CMP and content of MPP.

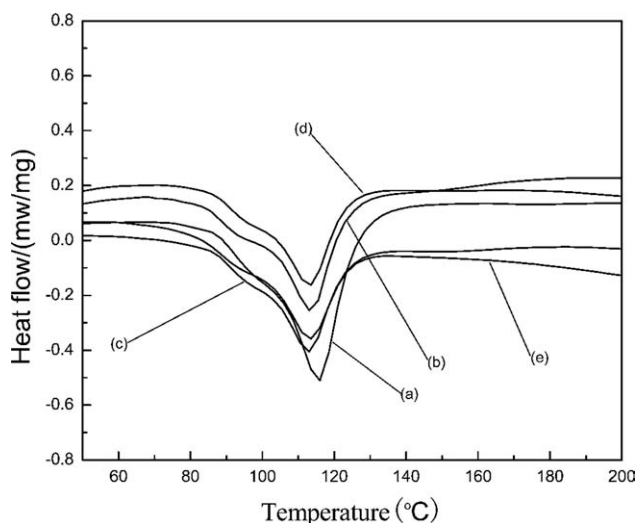


Figure 3 DSC curves of (a) LDPE, (b) LDPE/MPP/ST, (c) LDPE/MPP/ST/MH, (d) LDPE/MPP/ST/ATH, (e) LDPE/MPP/ST/FH.

comprising 20 wt % MPP reached up to 3.84, which is higher than those of the composites with other contents of MPP. The parameter indicated that MPP can reinforce resin matrix besides its fire resistance. The former research also indicated that the incorporation of organically modified montmorillonite and CaCO_3 improved both the tensile and thermal properties of composites.¹⁷

The results of flammability test were shown in Table I. It can be seen from Table I that the LOI values increased with the increase of MPP content. The composites comprising 20 wt % and 25 wt % MPP had a higher LOI compared with other contents of MPP. With the addition of MPP, the flame retardancy of LDPE was improved partly. The results corresponded to the report that flame retardation of ternary composites changed compared with binary composites.⁷ But it still caused a serious molten drops during the process of combustion for all composites. Because of possessing better fire resistance and mechanical properties (Fig. 2), composite comprising 20 wt % MPP was chosen to investigate the flame retardant properties of composites comprising different metal hydroxides.

It also can be seen from Table I that LOI increased slightly when composites comprising 5 wt % different metallic hydroxides. And all samples could not pass vertical burning test. LOI can further increase when the composites were added by 10 wt % different metallic hydroxides. The highest LOI of composites comprising different metallic hydroxides was 27% in these experiments, which represents an increase in 5 LOI units from the value of 22% that is the LOI of LDPE/MPP/ST system. And composite comprising 10 wt % ATH passed V1 rating test without causing molten drops. But the flame retardancy

of composite comprising 10 wt % MH or FH changed slightly. The main reason about these can be explained that composite comprising ATH formed thicker and more compact charred layer, which was more effective to isolate the O_2 in the air and protect the underlying and flammable substance of composites.

Thermal analysis

DSC analysis

The typical DSC curves could conclude whether there were crystal change or chemical reactions among LDPE and flame retardant system during the preparation of composites. Figure 3, which showed the DSC curves of LDPE and different composites. The DSC curves of all the samples possessed one phase change peaks near 120 °C which represented the solid-liquid phase change of the samples. Table II gave the relevant data obtained and derived from the DSC measurements, including peak temperature of melting (T_m), latent heat (enthalpy) in heating process (ΔH_m), initial transition temperature (T_i) and terminational transition temperature (T_t). It could be seen from Table II that no noticeable change in the melting peaks or the melting temperatures were detected among the samples except the ΔH_m and T_m of composites (b), (c), (d), and (e) decreased. The addition of MPP, ST, and metal hydroxides had no obvious effect on the crystallization of LDPE. In addition, sample (c), (d), and (e) (i.e., composites comprising different metallic hydroxides) had smaller value of latent heat in melting process (ΔH_m), T_m , T_i , T_t compared with sample (a) and (b). The possible explanation of this phenomenon was that the addition of metallic hydroxides limited the thermal molecular movements of LDPE. The research of Yibing Cai et al. also showed that the additives of flame retardant had little effect on the temperatures of phase change peaks and thermal energy storage property.¹⁸

TG analysis

The thermostability of pure LDPE is discussed and compared with that of different flame retardant

TABLE II
The Characteristic of Samples Obtained from DSC Analysis

Sample	T_m (°C)	ΔH_m (J/g)	T_i (°C)	T_t (°C)
a	115.6	-77.9	100.9	126.3
b	113.6	-56.0	99.3	123.5
c	112.8	-48.6	97.6	123.5
d	113.0	-42.3	98.1	123.4
e	113.3	-42.4	97.2	123.9

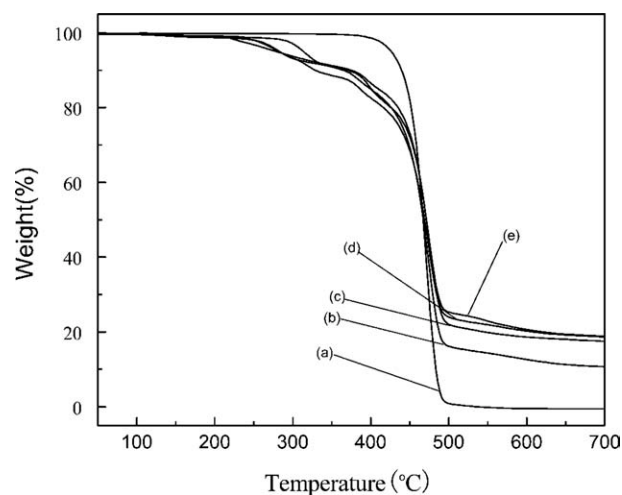


Figure 4 TG curves of (a) LDPE, (b) LDPE/MPP/ST, (c) LDPE/MPP/ST/MH, (d) LDPE/MPP/ST/ATH, (e) LDPE/MPP/ST/FH.

composites. The TG curves were presented in Figure 4. Table III reported the temperatures at 20% weight loss ($T_{0.2}$), temperatures at 50% weight loss ($T_{0.5}$), and weight residue at 450°C ($R_{450^\circ\text{C}}$) and 700°C ($R_{700^\circ\text{C}}$). In Figure 4, LDPE was found to undergo one-step degradation process. The addition of MPP, ST, and different metallic hydroxides induced the initiation of degradation process at lower temperature. It has been reported that the addition of IFR induced the initiation of degradation process at lower temperature.^{19,20} At the same time the degradation of all the composites appeared to begin at different temperature depending on the composition. As indicated in the Table III, both $T_{0.2}$ and $R_{450^\circ\text{C}}$ of all the composites (b, c, d, e) decreased compared with pure LDPE, whereas metallic hydroxides increased both $T_{0.5}$ and $R_{700^\circ\text{C}}$. The weight residue of (a) and (b) at 700°C were about 0 and 10%, respectively. But the weight residue of composites comprising 10 wt % different metallic hydroxides at 700°C was about 17–19%. Thus, the simultaneous incorporation of MPP and metallic hydroxide into the LDPE matrix was more effective than that of MPP alone. The reason may be explained that metallic hydroxide or its decomposition products react with the decomposition product of MPP to form phosphates or other organic

TABLE III
TG Data of the Samples

Sample	$T_{0.2}$ (°C)	$T_{0.5}$ (°C)	$R_{450^\circ\text{C}}$ (%)	$R_{700^\circ\text{C}}$ (%)
a	452	467	80.8	0
b	425	466	68.9	10.7
c	425	471	71.9	17.5
d	416	468	67.9	18.7
e	433	471	72.5	18.9

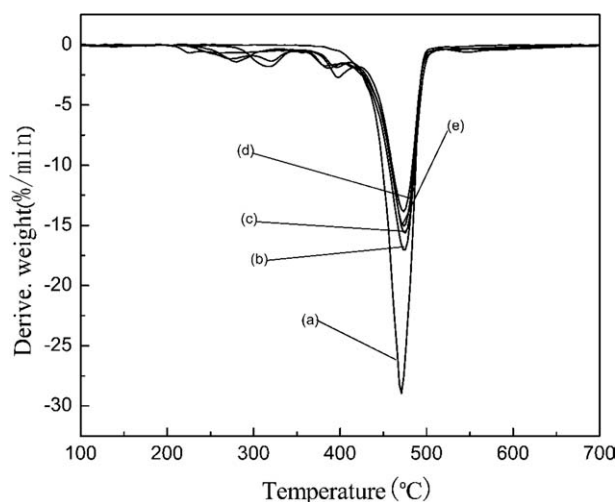


Figure 5 DTG curves of (a) LDPE, (b) LDPE/MPP/ST, (c) LDPE/MPP/ST/MH, (d) LDPE/MPP/ST/ATH, (e) LDPE/MPP/ST/FH.

produces, which could retard the circulation of combustible gas and promote the formation of charred layer.²¹ Furthermore, it is easy for the charred layer to accumulate on the surface of the composite and form a barrier layer for both underlying degradation products and incoming O_2 in the air.

DTG analysis

It can be seen from the DTG curves showed in Figure 5 that DTG has detected only one single peak for LDPE, which means that the degradation has happened in one-stage. And LDPE had the biggest rate of mass variation, whereas sample(d) had the smallest rate of mass variation. But all samples had the same temperature (about 480°C) reached maximum reaction rate. The analysis corresponded that LDPE/MPP/ST/ATH system possesses the best flame retardancy.

The DTG curves of (b), (c), (d), and (e) consisted of several partially overlapping peaks, indicating a multistep mechanism. The decomposition process could be divided into two stages according to the curves. At the first stage (about 100–415°C), the samples lost water and additives began the stage of oxidation and decomposition, which included both physical change and chemical change. At the second stage (about 420–700°C), it was the charring stage in which mainly were the decomposition of LDPE, MPP, metallic hydroxides, and the interreaction of all kinds of decomposition production. During this stage, the mass lost amounted to 80–90% (Fig. 4).

Morphology analysis

Figure 6 showed the surface morphology of residual charred layer of (a) LDPE/MPP/ST, (b) LDPE/

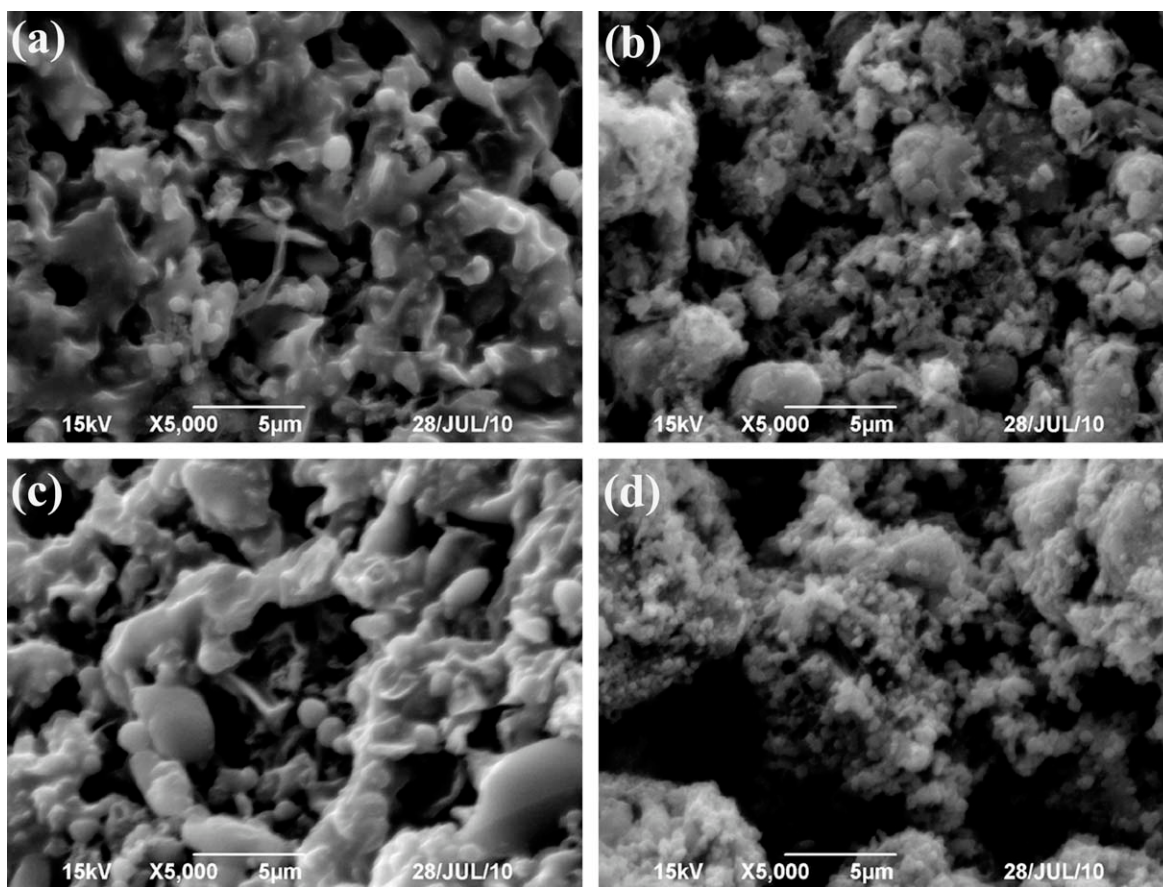


Figure 6 SEM photographs of residual charred layer of (a) LDPE/MPP/ST, (b) LDPE/MPP/ST/MH, (c) LDPE/MPP/ST/ATH, (d) LDPE/MPP/ST/FH.

MPP/ST/MH, (c) LDPE/MPP/ST/ATH, and (d) LDPE/MPP/ST/FH, respectively. From SEM photographs, it can be seen that there were remarkable differences in the morphology for all four composites. For LDPE/MPP/ST system [Fig. 6(a)], the accumulation of intumescent charred layer was very thin and some pores could be seen. For LDPE/MPP/ST/MH and LDPE/MPP/ST/FH system [Fig. 6(b) and six days], the charred layers were thicker than that of LDPE/MPP/ST and fewer pores could be seen on them. But the thickness of the charred layer was uneven and there were several big pores in some regions. In this sense, the uneven layer cannot endow the composites with good flame retardancy and play the roles of heat barrier and thermal insulator.^{22,23} Figure 6(c) showed the microscopic morphology of the LDPE/MPP/ST/ATH system. Comparing the charred layer with those of other composites, it is clear that the LDPE/MPP/ST/ATH system possessed the thickest charred layer and showed much more compact charred layer, which was even and double-decked. And the pores on it were smaller and fewer. The protective barrier limited the oxygen diffusion into the substrate and retarded the volatilization of the flammable

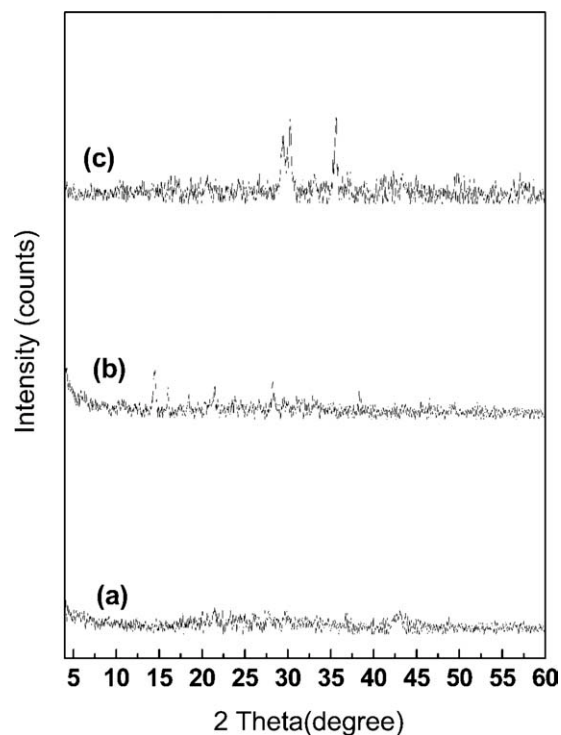


Figure 7 XRD patterns of residual charred layer of (a) LDPE/MPP/ST/MH, (b) LDPE/MPP/ST/ATH, (c) LDPE/MPP/ST/FH.

decomposition products. The morphology analysis corresponded to the flammability test that composite comprising 10 wt % ATH possessed the highest LOI and passed V1 rating test (Table I). It is obvious that the addition of metallic hydroxide could effectively improve the charred layer structure, hold back the combustion heat, and change the thermal stability.

XRD analysis

The XRD patterns of the residual charred layer of flame retardant composites were shown in Figure 7. In Figure 7, no obvious XRD peaks could be seen in pattern (a). It indicated that there was only a minute amount of crystalline solid in the residual charred layer in the LDPE/MPP/ST/MH composites.²⁴ In pattern (b), the XRD peaks at $2\theta = 14.4, 16.0, 18.4, 21.4,$ and 28.2° that correspond $d = 6.1, 5.5, 4.8, 4.1,$ and 3.2 nm were assigned to AlPO_4 , which formed via the conversion reaction between Al_2O_3 and thermal decomposition product of MPP. In pattern (c), three XRD peaks of the residue char at $2\theta = 30.2, 35.6,$ and 42.3° that correspond $d = 2.9, 2.5,$ and 2.1 nm were assigned to Fe_3O_4 . And several XRD peaks at $2\theta = 16.3, 20.6, 24.0, 29.5,$ and 33.0° that correspond $d = 5.4, 4.3, 3.7, 3.0,$ and 2.7 nm were assigned to $\text{Fe}(\text{PO}_3)_3$. Fe_3O_4 was the thermal decomposition product of FH. And the formed $\text{Fe}(\text{PO}_3)_3$ was also the reaction product of Fe_3O_4 and thermal decomposition product of MPP.²⁵ These reaction products could absorb some heat and retard oxidative reaction during the process of combustion. The mixture of these reaction products and amorphous carbon could form more stable charred layer which is good for the protection of underlying material of composites and endows the underlying material with a heat barrier.

CONCLUSIONS

According to the analysis of the results of experiments, it was evident that pure LDPE had low resistance to combustion and could not form any residual charred layer. The thermal behavior of LDPE/MPP/ST composite was improved by introducing metallic hydroxides. And composites comprising ATH were more stable than composites at the same additive loading with MH or FH, which was studied by LOI, TG, and DTG. The LOI of composite comprising ATH was found to increase from 22 to 27%. And composite comprising ATH passed V1 rating without causing molten drops. Furthermore,

DSC analysis showed that the addition of MPP, ST, and metallic hydroxides had no obvious effect on the crystallization of LDPE during the preparation of composites. It could be seen clearly from the SEM photo that the LDPE/MPP/ST/ATH system possessed the thickest charred layer, which was an even and double-decked charred layer. In addition, through XRD analysis of the residual charred layer, it found that AlPO_4 created via the conversion reaction between Al_2O_3 and thermal decomposition product of MPP. There was a synergy between certain metallic hydroxide and MPP.

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