

A Study of the Novel Intumescent Flame-Retarded PP/EPDM Copolymer Blends

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ABSTRACT: Flammability of polypropylene/ethylene-propylene-diene copolymer (PP/EPDM) filled with melamine phosphate (MP) and pentaerythritol phosphate (PEPA) was studied by limiting oxygen index (LOI), UL 94, and cone calorimetry. The thermal degradation of the composites was investigated using thermogravimetric analysis (TG) and real-time Fourier transform infrared spectrum (RT-FTIR), and the mechanical properties of the materials were also studied. It had been found that the PP/EPDM/PEPA/MP composites (PEPM series) showed

better flame retardancy than that of the PP/EPDM composites containing MP or PEPA. TG and RT-FTIR studies indicated that the interaction occurs among MP, PEPA, and PP/EPDM. The incorporation of the flame retardants deteriorated the mechanical properties of the materials. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 110: 3804–3811, 2008

Key words: PP/EPDM; intumescent flame retardation; melamine phosphate; pentaerythritol phosphate; thermal degradation

INTRODUCTION

Polypropylene (PP) as one of the major commodity polymers has excellent properties such as high stiffness and good mechanical properties. However, its brittle behavior limits the use of PP in many applications.¹ To improve the impact strength at low temperature, PP has usually been blended with different elastomers. Among these elastomers, ethylene-propylene-diene copolymer (EPDM) is commonly used because of its simple synthetic process and good weather resistance.^{2,3} Polypropylene/ethylene-propylene-diene copolymer (PP/EPDM) is extensively used in many fields; for example, cars, electric casings, interior decoration, insulation, and so on. Nevertheless, PP/EPDM blend is flammable, and the limiting oxygen index (LOI) of the blend is about 17.5%. This drawback restricts the range of its application. Therefore, it is important to study its flame retardation.

There are some publications on the flame retardation of PP/EPDM. For example, Li et al.⁴ have found

that the combination of decabromodiphenyl oxide and Sb₂O₃ improved the flame retardation of PP/EPDM greatly. However, halogen-containing flame-retarded polymers systems produce large amounts of smoke and toxic gases on burning. As for metal hydroxides, commonly used halogen-free flame retardants, their high loading levels (generally >60%) are needed to acquire an adequate flame retardant property, which will lead to a great decrease in the mechanical properties of filled polymer materials.^{5–7}

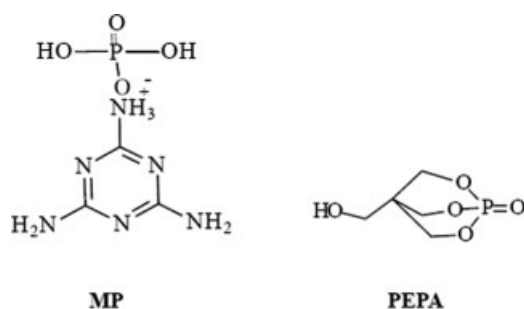
In the recent years, intumescent flame retardant (IFR) additives have aroused a great attention in the flame retardation of polymers. Intumescence is a strategy in flame retardancy, which involves in the formation on heating of a swollen multicellular thermally stable char insulating the underlying material from the flame action.⁸ A typical intumescent system comprises a dehydration catalyst for char formation [usually a phosphoric acid derivative, such as ammonium polyphosphate and melamine phosphate (MP)], a carbon rich polyol compound (e.g., pentaerythritol), and an organic amine or amide (i.e., melamine and polyamides) that is capable of producing noncombustible gases.⁹

MP, a reaction product of melamine and phosphoric acid, is a kind of typical blowing agent and acid source in the typical IFR system. It has been generally used in the flame retardation of polyamides.¹⁰ In our previous work, the flame retardation and the thermal degradation of PP/MP and PP/MP/PER series have been studied.¹¹ Pentaerythritol phosphate (PEPA), that is, 2,6,7-trioxa-1-phosphabicyclo

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Scheme 1 Structure of MP and PEPA.

[2,2,2] octane-4-methanol 1-oxide, can be used as carbonization agent and acid source in the IFR system. PEPA, used as a flame retardant, has been reported in glass fiber-reinforced PET, a PPO/HIPS blend, and epoxy resins.^{12–15} However, the use of MP or PEPA individually does not comprise a typical intumescent system according to the definition of intumescent strategy and thus cannot achieve excellent fire-retardant properties in polyolefins.

In this study, the combustion characteristics of flame-retarded PP/EPDM composites containing PEPA, MP, and PEPA/MP mixture are studied using LOI, UL 94, and cone calorimetry. The thermal stability and degradation properties of the composites are investigated by thermogravimetric analysis (TG) and RT-FITR. The mechanical properties of the materials are also studied.

EXPERIMENTAL

Materials

PP (F401) was provided by Yangzi Petroleum Chemical Company, China. Ethylene-propylene-diene copolymer (EPDM 5565) containing 50 wt % ethylene and 7.5 wt % ethylidene norbornene was supplied by DuPont Dow Elastomer, USA. MP was supplied by Hefei Jinhui Institute of Chemical Engineering, China. PEPA was provided by BaLin Petroleum Chemical Company, China. MP and PEPA were dried at 100°C for 24 h before use. The structures of MP and PEPA were shown in Scheme 1. The d_{50} value for the particle size distribution of MP and PEPA was 61.3 and 62.0 μm , respectively.

Preparation of samples

The preparation of flame-retarded PP/EPDM composites was done on a Brabender-like apparatus. After the PP is melted, EPDM was added into the apparatus, mixed them for about 5 min. After EPDM was dispersed into PP uniformly, the flame retardants were added into the PP/EPDM blends (the formulations are given in Table I). The resulting mixture was

mixed at a temperature range of 170–175°C for 10 min. After mixing, the samples were hot pressed at about 175°C under 10 MPa for 5 min into sheets of suitable thickness and size for analysis. The weight ratio of PP/EPDM in all flame-retarded PP/EPDM composites and the untreated PP/EPDM was 3 : 2.

Characterization

Limiting oxygen index

Limiting oxygen index (LOI) was measured according to ASTM D2863. The apparatus used was an HC-2 oxygen index meter (Jiangning Analysis Instrument Company, China). The specimens used for the test were of dimensions 100 × 6.5 × 3 mm.

UL94 vertical burn tests

The vertical test was carried out on a CFZ-2-type instrument (Jiangning Analysis Instrument Company, China) according to the UL 94 test standard. The specimens used were of dimensions 130 × 13 × 3 mm.

Cone calorimetry

The cone calorimeter (Stanton Redcroft, UK) tests were performed according to ISO 5660 standard procedures. Each specimen of dimensions 100 × 100 × 3 mm was wrapped in aluminum foil and exposed horizontally at an external heat flux of 35 kW/m².

Thermogravimetry

Each sample was examined under air on a STA 409°C TGA apparatus (Netzsch Company, Germany) with crucible sample holders, at a heating rate of 10°C/min.

Real-time FTIR spectroscopy

Real-time Fourier transform infrared (RT-FTIR) spectra were recorded using a Nicolet MAGNA-IR 750 spectrophotometer equipped with a ventilated oven having a heating device. The untreated PP/EPDM

TABLE I
Formulations of Flame-Retarded PP/EPDM Composites

Sample code	PP/EPDM	MP (phr)	PEPA (phr)
PE	100	0	0
PEM1	100	18	0
PEM2	100	42	0
PEM3	100	66	0
PEP	100	0	66
PEPM1	100	24	42
PEPM2	100	33	33
PEPM3	100	49.5	16.5

TABLE II
LOI Values and UL 94 Testing Results of
Flame-Retarded PP/EPDM Composites

Sample code	LOI (%)	UL-94 rating
PE	17.5	No rating
PEM1	21	No rating
PEM2	25	No rating
PEM3	26	No rating
PEP	28.5	V-1
PEPM1	28.5	V-0
PEPM2	30	V-0
PEPM3	32.5	V-0

(sample PE) and flame-retarded PP/EPDM composites (sample PEPM2) were mixed with KBr powders, and the mixture was pressed into a tablet, which was then placed into the oven. The temperature of the oven was raised at a heating rate of about 10°C/min. Dynamic FTIR spectra were obtained *in situ* during the thermal oxidative degradation of the polymer and its composites.

Mechanical properties

The tensile strength and elongation at break were measured with a WD-20D Electronic Universal Testing Machine at the crosshead speed of 500 mm/min.

RESULTS AND DISCUSSION

LOI and UL 94 rating

Table II presents the LOI values and UL-94 test results of the flame-retarded PP/EPDM composites. As for the PEM series composites, it can be seen that the LOI values of the composites increase with the increase of MP content. The LOI value of the composite containing 40 wt % MP was 26%. However, there are no ratings for the PP/EPDM/MP composites at all, even at the MP loading of 40 wt %. This illustrated that MP used alone in PP/EPDM did not have good flame retardancy. When PEPA was incorporated into the PP/EPDM/MP composites, a remarkable improvement of flame retardation was observed. It was clear that the LOI values of PEPM

series were higher than that of PEM series. Moreover, all sorts of the PEPM series composites could reach the V-O rating in UL-94 test. As seen from Table II, the PP/EPDM composites containing only PEPA cannot have good flame retardancy. Although the PEPA loading was as high as 40 wt %, the PP/EPDM/PEPA composite just reached the UL-94 V-1 rating.

Cone calorimeter studies

Although small scale tests such as LOI and UL 94 are widely used in realizing and evaluating flame retardance in polymers, they are not reliable indicators of likely performance in a real fire. For this kind of analysis, cone calorimetry is becoming the method of choice, and it can provide a wealth of information on the combustion behavior of materials.¹⁶ Relevant cone calorimetry parameters of the flame-retarded PP/EPDM composites were given in Table III.

Heat release rate (HRR), in particular the peak HRR (PHRR), has been found to be one of the most important parameters to evaluate fire safety. Figure 1 shows the RHR curves of flame-retarded PP/EPDM composites and the untreated PP/EPDM obtained from the cone calorimeter test. It can be seen that the material without flame retardant burns very fast after ignition, and a sharp RHR peak appears with a PRHR value of 1302 kW/m². In the case of the PEM series composites, their PRHR values were greatly reduced and decrease with increasing the MP content, as shown in Figure 1. Moreover, the combustion time of the PP/EPDM /MP composites was prolonged in comparison with that of the untreated PP/EPDM.

When PEPA was added into PP/EPDM /MP composites, a significant decrease of the PRHR of the flame-retarded polymers was observed. As shown in Figure 1, when PEPA : MP ratio was 3 : 5, the PRHR was only 217 kW/m². The possible explanation was that the efficient and stable intumescent char formed during the burning process of the composites PP/EPDM/PEPA/MP, which can prevent the heat and mass transfer between the flame zone and the burning substrate, protect the underlying materials from

TABLE III
Cone Calorimeter Data of Flame-Retarded PP/EPDM Composites

	PE	PEM1	PEM2	PEM3	PEP	PEPM1	PEPM2	PEPM3
Time to ignition (s)	57	36	37	43	39	42	50	35
Peak RHR (kW/m ²)	1302	572	429	383	351	217	291	262
Average RHR (kW/m ²)	514	258	252	137	122	64	115	123
Average SEA (m ² /kg)	644	785	734	594	949	345	518	799
Average CO (kg/kg)	2.80	2.29	1.96	1.98	1.67	1.89	1.97	1.78
Average CO ₂ (kg/kg)	0.036	0.040	0.035	0.026	0.086	0.049	0.055	0.071
Fire performance index	0.043	0.062	0.086	0.12	0.11	0.19	0.17	0.12

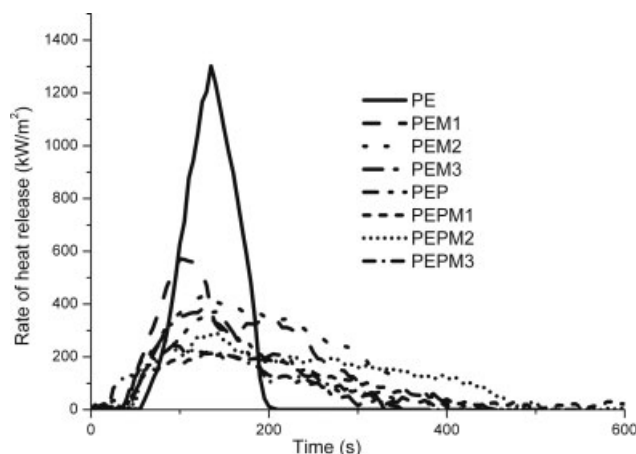


Figure 1 RHR curves of flame-retarded PP/EPDM composites versus the untreated PP/EPDM under a heat flux of 35 kW/m^2 .

further burning and retard the pyrolysis of polymers, and thus result in a great decrease in PRHR. As can be seen from Figure 1, the combustion time of the PEPM series was longer compared with that of the PEM series. PEPA as carbon and acid sources was in conjunction with MP would make the intumescent coating more stable and accordingly delay the combustion time of the material.

It is noted from Table III that the time to ignition (TTI) of both the PEM series and PEPM series was lower than that of the untreated PP/EPDM. This was a typical characteristic of intumescent flame-retardant systems and had been previously found by other researchers.^{17,18} The reason for the decrease in TTI may be due to the decomposition of intumescent flame retardants or the accelerated decomposition of the polymers induced by the flame retardants. The detailed reason was still unclear. However, HRR or TTI used only cannot reflect the risk of fire during the combustion process of the material comprehensively. At present, fire performance index, that is, the value of TTI /PRHR, had been thought to be a relative reliable parameter to evaluate the potential flammability of the material.^{19,20} Under the same heat flux, the higher the value of fire performance index was, the lower the potential of the material to burn. As shown in Table III, the fire performance index values of PEM series increased with the increasing amounts of MP. Moreover, the fire performance index values of PEPM series were higher than that of PEM series, illustrating that the combination of PEPA with MP could make the flame-retarded material much safer in a fire.

The concentration of CO released during the burning course of the material was another key factor to estimate the hazard of fire. The bigger the concentration of CO released, the more dangerous the smoke

will become. The temporal behavior of the CO evolution rate during the cone calorimetry experiments for flame-retarded PP/EPDM composites and the untreated PP/EPDM was shown in Figure 2. In the case of PEM series, the average values of CO concentration decreased with the increasing amounts of MP. As for PEP series, with the increase of PEPA loadings, the values rose. This may be explained by the following reasons. Phosphoric-containing acid formed during the decomposition process of PEPA could cover the surface of the material, which hindered the contact between oxygen and underlying material, and thus lead to the incomplete burn of the material. Moreover, the second CO peak appeared during the last part of the combustion process, which could be caused by the incomplete combustion of the formed intumescent charred layer.

As for the smoke emission from the burning of the PP/EPDM /MP composites, it can be seen from Table III that their average-specific extinction area (ASEA) values decreased as the MP content increased, which illustrated that MP was not only a RHR reducer, but also had a smoke suppression effect. The ASEA values of the PEPM series were lower than that of PEM series, meaning that the combination of PEPA with MP can reduce the emission of smoke further.

Thermogravimetric analysis

Figure 3 showed the thermogravimetric (TG) curves of MP, PEPA, and the 50 wt % MP/50 wt % PEPA mixture. MP began to decompose at about 240°C to form melamine pyrophosphate, which further decomposed to form melamine polyphosphate, melam ultraphosphate, etc., as proposed by Camino and Costa.⁸ It lost weight very gradually and left about

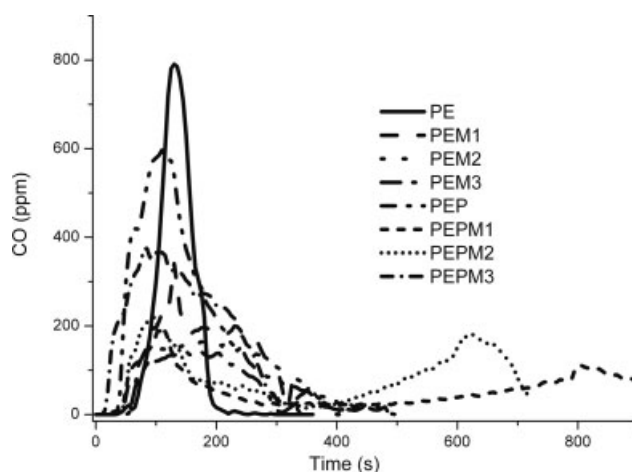


Figure 2 CO versus time curve of flame-retarded PP/EPDM composites versus the untreated PP/EPDM under a heat flux of 35 kW/m^2 .

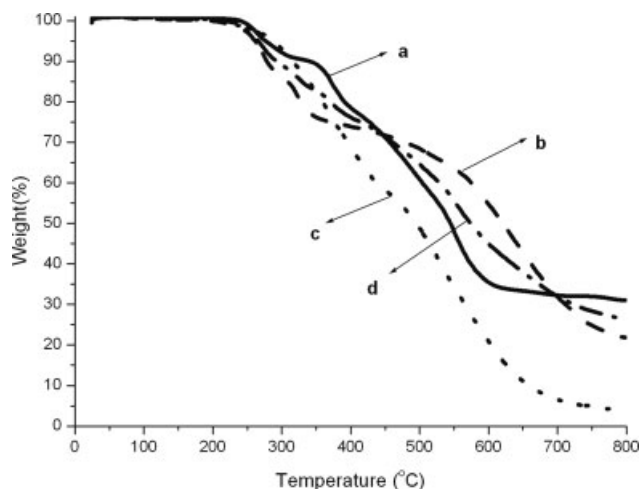


Figure 3 TG curves: (a) MP; (b) PEPA; (c) and (d) 50%MP/50%PEPA mixture (experimental and calculated, respectively) in the air atmosphere.

30 wt % residue after the decomposition at 800°C. The decomposition of PEPA mainly consisted of three steps of weight loss. It started to decompose at about 210°C with the dehydration of PEPA. The second step occurred in the range of 300–360°C. PEPA opened its cyclic structure gradually and then condensed to pyrophosphate simultaneously. The percentages of weight loss of the two steps were 16 wt % and 14 wt %, respectively. A 54 wt % weight loss was observed during the third stage happening above 360°C. In this stage, the carbonaceous char was formed with the elimination of H_3PO_4 , and pyrophosphate was likely to decompose partly, resulting in the big weight loss in this stage. About 21 wt % solid residue left at 800°C.

The thermal decomposition behavior of the mixture of MP and PEPA differed from that as expected on the basis of additive behavior of MP and PEPA. This was well demonstrated by comparing the experimental and calculated thermograms. According to the literature, it was representative of a noninteracting behavior among the components if the calculated curve was a linear combination of the TG curves of the individual components of the system.²¹ As shown in Figure 3, the mixture (experimental curve) was more thermally stable before 350°C compared with that of the calculated one, but it had a more rapid stage of weight loss above 350°C and produced less amounts of residue at 800°C than that of the calculated. This result showed that there was an interaction between MP and PEPA during the thermal decomposition.

Figure 4 showed the TG curves of samples PE and PEPM2. After 247°C, sample PEPM2 were more thermally stable than the untreated PP/EPDM. The amount of residue of sample PEPM2 at 600°C was

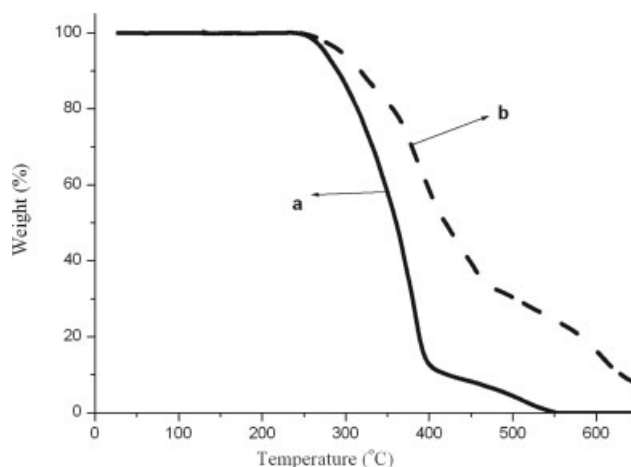


Figure 4 TG curves: (a) sample PE; and (b) sample PEPM2 in the air atmosphere.

about 16.3 wt %, while sample PE did not leave any char residue at the temperature. This could be caused by the good stability of a carbonaceous material formed during the decomposition process of sample PEPM2.²² It was clear that the incorporation of the flame retardants significantly enhanced the thermal stability of PP/EPDM.

Thermal degradation studies

The thermal degradation behavior of the sample PE and PEPM2 was investigated using RT-FTIR spectroscopy. Figures 5 and 6 showed the changes in the dynamic FTIR spectra obtained from the samples PE and PEPM2 at different pyrolysis temperatures.

As shown in Figure 5, at 25°C, sample PE had three kinds of absorptions, that is, C—H, C—C and C=C. The peaks at 2970 cm^{-1} and 2845 cm^{-1} were the typical symmetrical and asymmetrical absorptions of C—H group. The peaks at 1633 cm^{-1} and 1166 cm^{-1} were assigned to the absorptions of C=C and C—C, respectively. It could be seen from Figure

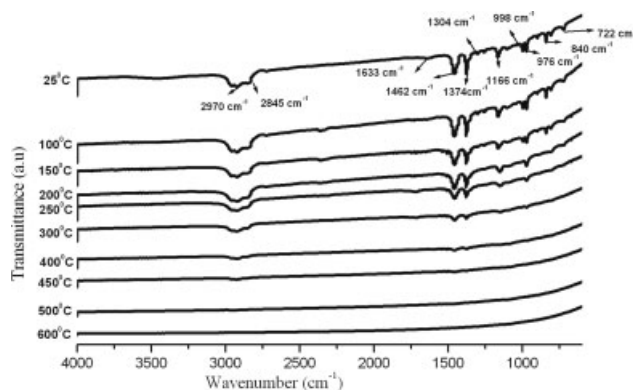


Figure 5 Dynamic FTIR spectra of sample PE with different pyrolysis temperatures.

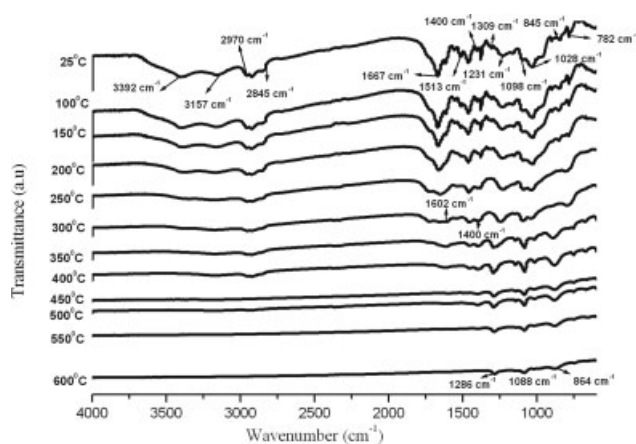


Figure 6 Dynamic FTIR spectra of sample PEPM2 with different pyrolysis temperatures.

5 that the relative intensities of the characteristic peaks changed negligibly below 150°C. As the temperature reached 250°C, the relative intensity of C—H absorption peaks around 2900 cm^{-1} and the peak at 722 cm^{-1} , ascribed to the typical absorption of CH in $-(\text{CH}_2)_n-$ linkage ($n > 4$), decreased quickly, which illustrated the degradation of the polymer resin.²³ These peaks disappeared at 500°C eventually, meaning that the polymer decomposes completely.

Figure 6 shows the RT-FTIR spectroscopy of the sample PEPM2. The relative intensity of the peaks at 3392 cm^{-1} (stretching absorption of O—H) decreased gradually from 150°C and disappeared at 350°C. This could be caused by the dehydration of MP and PEPA.²⁴ At 25°C, the peaks at 1309 and 1231 cm^{-1} were attributed to the O=P absorption of PEPA and MP, respectively. The O=P absorption of MP was at relative lower wave numbers because of hydrogen bonding. As shown in Figure 6, the relative intensity of the peak at 1028 cm^{-1} (P—O—C stretch) decreased gradually from 200°C, resulting from the degradation of the group of PEPA. This is in agreement with the change of the peak at 845 cm^{-1} , which was assigned to the characteristic absorptions of cyclic structure of PEPA.^{24,25} At 250°C, PEPA began to decompose to form unsaturated phosphorous-containing acid, which further decomposed to form carbonaceous char.²⁵ The related possible reaction mechanism was shown in Scheme 2.

The peaks at 3157 cm^{-1} and 1667 cm^{-1} , assigned to the symmetric stretching and deformation of N—H, respectively, decreased quickly from 200°C and disappeared by further heating to 300°C. When the temperature was up to 300°C, the new peak at 1602 cm^{-1} appeared, and it disappeared at 450°C. The peaks at 1513 and 782 cm^{-1} , attributed to the characteristic absorptions of triazine group, disap-

peared at 250°C. These transformations could be caused by a progressive condensation of the melamine groups with the elimination of NH_3 . According to the literature, at above 250°C, melamine was condensed to melam, melem, and melon with the increase of the temperature (as shown in Scheme 3).⁸

It was noted that the intensity of the peak around 1400 cm^{-1} decreased first from 250°C and increased at above 350°C. Then, it disappeared at 550°C. At 25°C, the peak was assigned to the absorption of ammonium ions, and its intensity decreased from 250°C because of the elimination of melamine from ammonium salt complexes.¹¹ The intensity of the peak increased at above 300°C could be attributed to the formation of phosphorous oxynitrides. At above 300°C, the unsaturated phosphorous-containing acid formed by the decomposition of PEPA was likely to react with the amino groups (melam, melem, and melon) to form phosphorous oxynitrides.²⁶

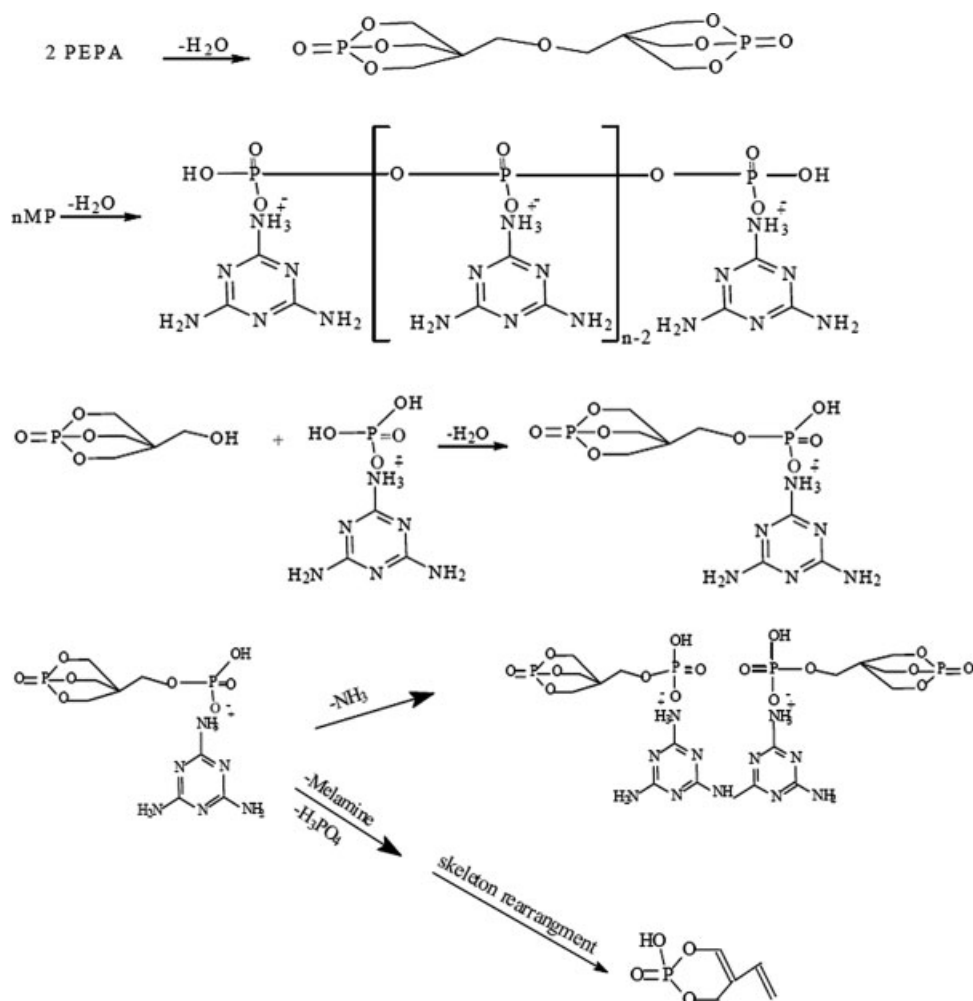
At 600°C, there are three peaks (1282 cm^{-1} , 1088 cm^{-1} , and 886 cm^{-1}) as shown in the spectrum, indicating that the residue was composed of P=O and P—O—P groups. It could be concluded that some complicated reactions had occurred during the thermal degradation process of sample PEPM2, meanwhile, many noncombustible gases and phosphorous-containing acids formed, improving the flame retardancy of the material.

Mechanical properties

Table IV shows the mechanical properties of the flame-retarded PP/EPDM composites. It was expected that the tensile strength and elongation at break decrease with increasing the amount of flame retardants. For example, when MP or PEPA loading increased to 40 wt % (samples PEM3 and PEP), their tensile strengths decreased to 5.9 and 10.8 MPa, and elongations at break reduced to 19.6 and 18.9%, respectively. The tensile strength and elongation at break also decreased when the PEPA/MP mixtures were added into the PP/EPDM blends. The above results indicated that the mechanical properties of the flame-retarded PP/EPDM composites were deteriorated by the addition of fillers MP and PEPA, which could be caused by that the fillers MP and PEPA had a high d_{50} -value for the particle size distribution.

CONCLUSION

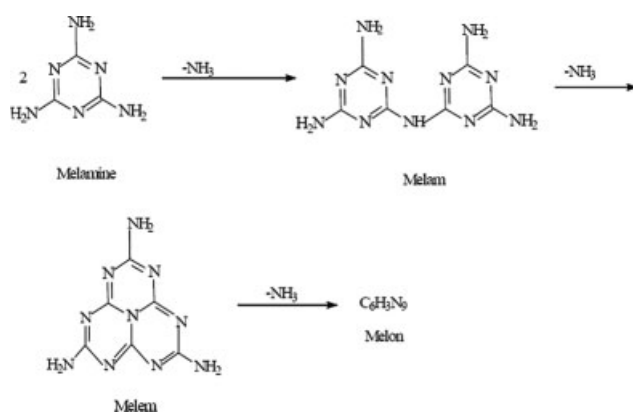
It was found that MP or PEPA when used alone in the PP/EPDM blends produced a little improvement in the flame retardation. The combination of MP and PEPA shows more effective flame retardation than the individual component. All composites containing both MP and PEPA had a high LOI value and



Scheme 2 The related possible reaction mechanism.

reached the UL-94 V-O rating. Cone calorimeter results demonstrated that MP in combination with PEPA can help to reduce the HRR and smoke emission. TG studies proved that the PP/EPDM/MP/PEPA composites were more thermal stable than the

untreated PP/EPDM. It was observed from the RT-FTIR study that many complicated reactions would take place during the thermal degradation process of the PP/EPDM/PEPA/MP composites. Moreover, the mechanical properties of the PP/EPDM composites were deteriorated with addition of the flame retardants.



Scheme 3 Possible condensation process of melamine.

TABLE IV
Mechanical Properties Measurements Results of
Flame-Retarded PP/EPDM Composites

Sample code	Tensile strength (MPa)	Elongation at break (%)
PE	13.6	331
PEM1	10.5	153
PEM2	7.4	63
PEM3	5.9	20
PEP	10.8	18
PEPM1	10.9	32
PEPM2	10.2	21
PEPM2	10.2	17

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