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Flammability Performance of Intumescent Flame Retardant EPDM/PP-Based Thermoplastic Elastomers†

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Flammability performance of intumescent flame retardant (IFR) ethylene-propylene-diene-terpolymer (EPDM)/polypropylene (PP) thermoplastic elastomers combined with montmorillonite (MMT) were evaluated using limiting oxygen index (LOI) and vertical combustion tests. The influences of montmorillonite on microstructure of the residual chars were studied by scanning electron microscope. The synergy between intumescent flame retardant and montmorillonite in improving flame retardancy of the materials has been found.

Key Words: Thermoplastic elastomers, Intumescent flame retardant, Montmorillonite.

INTRODUCTION

Thermoplastic elastomers (TPEs) have attracted considerable technologically interest because they combine the physical properties of elastomers with the process ability of thermoplastic polymers. The most used TPEs are blends of polyolefin, such as polypropylene (PP) with ethylene-propylene-diene-terpolymer (EPDM) rubber[1,2]. However, due to their chemical constitution, TPEs are inherent flammable and flame retardancy becomes an important requirement for the materials.

The fire protection of polymeric materials by intumescent flame retardants (IFR) is a typical condensed-phase flame retardant mechanism[3,4]. The intumescent char will develop a foamed carbonaceous shield under heat flux to retard the transfer of oxygen and heat as well as reduce the escape of volatile. Meanwhile, it is known that the formation of nanostructure in polymer matrixes by the introduction of clay, such as montmorillonite (MMT), will impart better fire resistance to the nanocomposites[5,6].

In this paper, IFR flame retardant EPDM/PP-based TPEs were prepared and the synergy between IFR and MMT in enhancing flammability performance of the materials was investigated.

EXPERIMENTAL

Flame retardant EPDM/PP-based thermoplastic elastomers (TPEs) were melt compounded of EPDM/PP (60/40 by weight) with desired amount of flame retardants, melamine phosphate (MP) and pentaerythritol (PER), in a Brabender mixer at 180 ºC for 10 min, using 5 phr (part of reagent per hundred of EPDM) SP-1045 (phenol-formaldehyde resin) in combination with 1 phr $SnCl_2$ and 1.5 phr ZnO as curing agent systems. To obtain clay-containing TPE/IFR samples, PP/MAPP (maleated polypropylene)/OMT (hexadecyl trimethyammounium modified montmorillonite) (85/15/10 by weight) masterbatch was firstly extruded using a twin-screw extruder at 160-180 ºC. The strands were then pelletized and mixed with EPDM and flame retardant additives in the mixer for 10 min. The resulting mixtures were compress-molded into 3 mm-thick sheets. The formulations are shown in Table-1.

Fire testing and SEM studies: Limiting oxygen index (LOI) determinations were performed with a sample of dimensions 100 mm × 6.5 mm × 3 mm according to ASTM D2863. UL-94 vertical burning tests were performed with a plastic sample of dimensions 130 mm × 13 mm × 3 mm, suspended vertically above a cotton patch. Five specimens were taken to estimate the precision of the reported data. The scanning electron microscopy (SEM) images of the char after LOI experiments were observed using a Hitachi X650 scanning electron microscope.

RESULTS AND DISCUSSION

Combustion behaviour: As listed in Table-1, one can see that the incorporation of IFR into TPE increases the LOI

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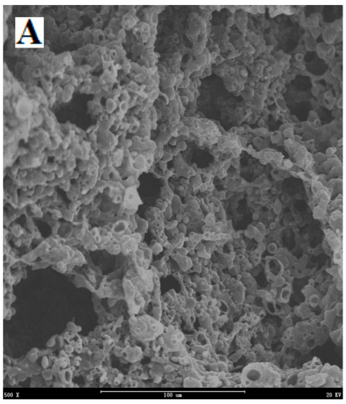
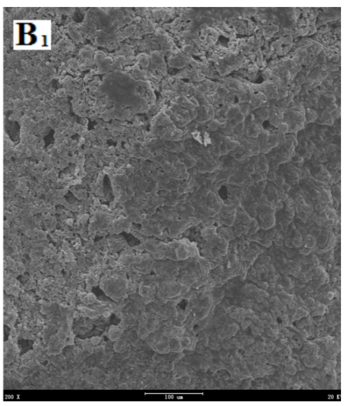
TABLE-1
COMPOSITION AND FLAMMABILITY PERFORMANCE OF TPES

Sample code	Composition (g)				LOI	UL-94
	EPDM	PP	PP/ MAPP/ OMT	MP/PER (2.5/1 by weight)		
TPE	60	40	–	–	–	–
TPE-IFR-1	60	40	–	55	29.5	Fail
TPE-IFR-2	60	40	–	60	30.0	Fail
TPE-IFR-3	60	40	–	65	30.5	V-0
TPE-IFR/clay-1	60	–	40	55	28.5	Fail
TPE-IFR/clay-2	60	–	40	60	29.0	V-0
TPE-IFR/clay-3	60	–	40	65	29.5	V-0

values slightly and UL 94 V-0 classification is achieved at TPE-IFR-3 at about 38 wt % IFR loading. Combination of clay with IFR did not impart TPE/IFR/clay samples with higher flame retardancy in terms of LOI. However, TPE-IFR/clay-2 sample achieves V-0 classification in the UL-94 test, which gives positive evidence that a synergistic effect occurred when IFR and clay are both present. It is known that exudation and water solubility are shortcomings of PER, which is often incompatible with the polyolefins. It is therefore expected that the materials with lower global IFR loading will avoid these problems and impart better mechanical properties to the materials.

Morphology of residual char: Both the physical and chemical properties of the intumescent char have important roles on the fire performance of polymeric materials. Fig. 1 shows the photographs of the chars collected from TPE-IFR-2 and TPE-IFR-3. TPE-IFR-2 (Fig. 1A) leaves an intumescent char composited of numerous incompleted foamed cellulae and a few holes in the char can been found. In the case of TPE-IFR-3 (Fig. $1B_1$ and $1B_2$), a tighter char is formed and most of the cellulae are completed, which provides the substrate better fire protection.





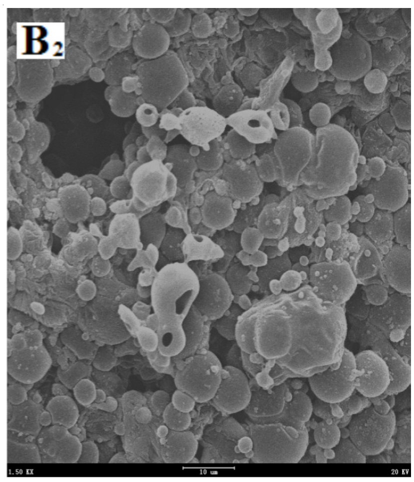
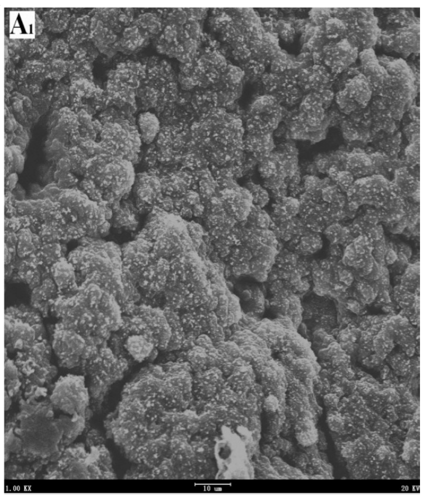


Fig. 1. SEM micrographs of the char form (A) TPV-IFR-2 and (B_1, B_2) TPV-IFR-3 at different magnification

TPE-IFR/clay-2 shows different residues in appearance compared to TPE-IFR samples and its external and interior structures of the char are shown in Fig. 2. As shown in Fig. 2(A_1 and A_2), an integrated char covering the sample surface is observed. Mean while, numerous white particles are beset on the surface. This is maybe caused by the migration of the MMT particles within the polymeric matrix and the aggregation on the surface. The interior structure of the char shown in Fig. 2 (A_3) is denser than that either TPE-IFR-2 or TPE-IFR-3, which may be attributed to the formation of ceramic-like material with high mechanical properties by the incorporation of MMT into IFR. It is therefore expected that this char structure can provide dual protection on the material from the heat and improve the material fire retardancy.



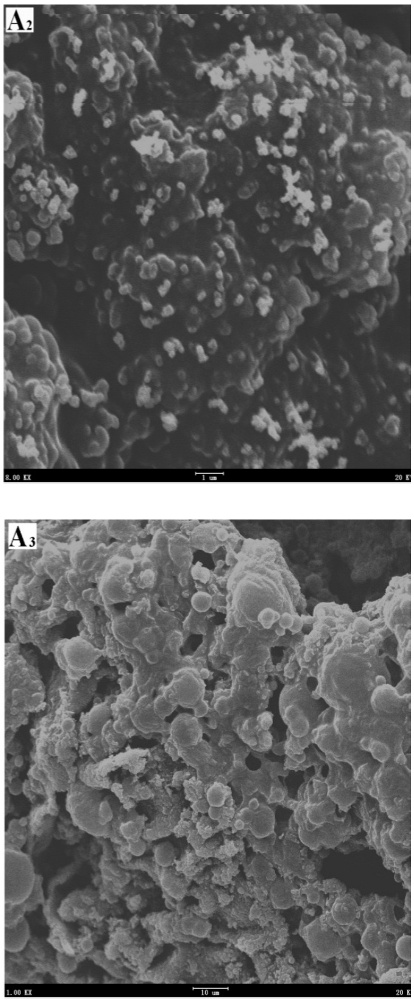


Fig. 2. SEM micrographs of the char form TPE-IFR/clay-2 (A_1 and A_2) external structure at different magnification and (A_3) interior structure

Conclusion

Intumescent flame retardant TPE composites prepared using phenolic curing agent in combination with stannous chloride and zinc oxide as vulcanizing systems have been investigated. Improvement in fire performance of TPE/IFR composites by the introduction of MMT has been reported. Compared to TPE/IFR composites, the char from TPE/IFR/MMT with structure in which the intumescent foams in addition to the accumulation of MMT particles over the substrate is efficient to stabilize the barrier.

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