

# Optimum Compatibilization for the Nonflammability of Thermoplasticized Crosslinked Polyethylene/Metal Hydroxides Composites with a Compatibilizer

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**ABSTRACT:** Nonflammability of the thermoplasticized crosslinked polyethylene (PE)/polyolefin elastomer (POE)/metal hydroxide flame retardant/compatibilizer composites were investigated. The thermoplasticized crosslinked PE was decrosslinked from the crosslinked high-density PE under the supercritical methanol condition. Two types of metal hydroxides: aluminum hydroxide (AH) and magnesium hydroxide (MH), and a low-density polyethylene-g-maleic anhydride (LM) were used as flame retardants and a compatibilizer, respectively. Nonflammability of both PE/POE/AH/LM and PE/POE/MH/LM composites was enhanced with metal hydroxide flame retardant concentration. PE/POE/MH/LM composites had better nonflammability than PE/POE/AH/LM composites at the same flame retardant concentration. Interestingly, nonflammability of the compo-

sites was also strongly influenced by the compatibilizer concentration. At low compatibilizer concentration, the nonflammability of the PE/POE/MH/LM composites was improved with the compatibilizer concentration. In contrast, at high compatibilizer concentration, the nonflammability of the PE/POE/MH/LM composites was deteriorated with the compatibilizer concentration. This demonstrates that optimum concentration of the compatibilizer can help to enhance the efficiency of the flame retardants in the development of the nonflammable polyolefin for the wire and cable industry. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 124: 2814–2823, 2012

**Key words:** nonflammability; flame retardant; metal hydroxide; compatibilizer; decrosslink; supercritical; polyethylene

## INTRODUCTION

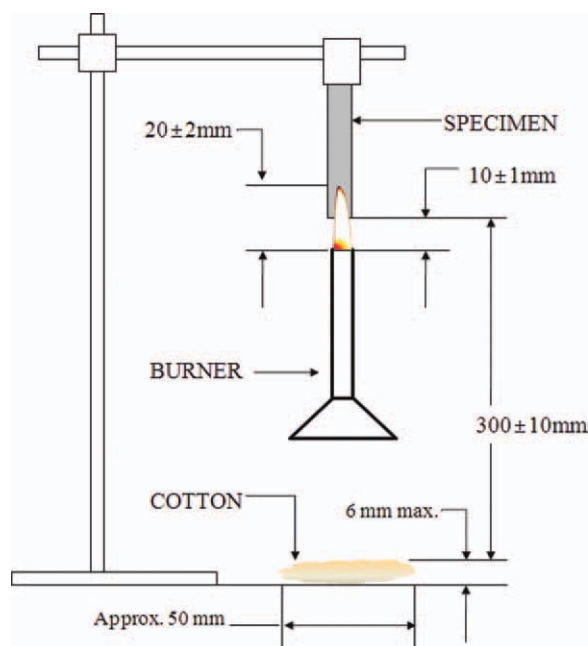
Recycling of thermosetting polymers such as crosslinked polyethylenes (PE) has garnered great attention owing to the deep consideration given to environmental pollution and to resource conservation efforts around the world.<sup>1–6</sup> An eco-friendly supercritical fluid process was introduced as a recycling technique to transform the crosslinked PE into thermoplasticized PE. In a previous article, the authors developed a kinetics model for the thermoplasticization reaction of crosslinked PE based on experimental results in which the decrosslinking reaction rate was linearly proportional to the gel concentration and related exponentially to the temperature.<sup>4</sup>

The PE are widely used in the wire and cable industry due to their good mechanical and physical

properties, as well as their easy processing. However, PE have some disadvantages such as high flammability and poor compatibility with additives such as flame retardants.<sup>7</sup>

The nonflammability of the PE is usually enhanced by flame retardants such as halogen-containing compounds and metal hydroxides.<sup>7–11</sup> Despite their high effectiveness in flame retardancy, the conventionally used halogen-containing flame retardants have been avoided because of their problems of toxicity, corrosion, and smoke. In contrast, metal hydroxides have taken increasing attention owing to their desirable combination of low cost, low smoke, and relatively high flame retardant efficiency.<sup>7–9</sup> However, a high loading of metal hydroxides is needed to obtain an adequate level of nonflammability, and poor adhesion between PE and metal hydroxides occurred.<sup>7,8</sup> These factors lead to a reduction of mechanical properties such as elongation at break and toughness. These problems could be overcome by adding polyolefin rubber and

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**Figure 1** Illustration of the UL94 vertical burning test setup according to ASTM D3801. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

polar group functionalized compatibilizers to the compounds.<sup>10</sup>

In this work, to improve the physical properties and nonflammability, the thermoplasticized crosslinked PE composites with metal hydroxide flame retardants and a compatibilizer are demonstrated. The PE was decrosslinked from the crosslinked PE with 88 wt % gel fraction using supercritical methanol. Polyolefin elastomers (POE) were used as an additive to enhance the toughness of the composites. Two types of metal hydroxides (aluminum hydroxide (AH) and magnesium hydroxide (MH) and a low-density polyethylene-*g*-maleic anhydride (LM) were used as flame retardants and a compatibilizer, respectively. The effect of the compatibilizer and metal hydroxide flame retardants on the nonflammability of the polyolefin composites is investigated with a consideration of the reinforcement effect of the filler and compatibilizer, and of the adhesion properties. Interestingly, the nonflammability of the polyolefin composites with metal hydroxide flame retardants is strongly influenced by the compatibilizer concentration.

## EXPERIMENTAL

### Materials

The crosslinked high-density PE were supplied by Mikwang Chemical in South Korea. The crosslinked PE had a gel fraction of 88%. Methanol (99.5%, Samchun Chemical) was used as supercritical media.

POE with the trade name LC170 was supplied by LG Chemicals. The density and melt flow index of the POE were 0.870 g/cm<sup>3</sup> and 1.1 g/10 min according to ASTM D1238. A linear low-density polyethylene-*g*-maleic anhydride with a trade name of LE149V (LM), provided by Hyundai Engineering Plastic, was used as a compatibilizer. The density and melt flow index of the LM were 0.921 g/cm<sup>3</sup> and 1.6 g/min. Two types of metal hydroxides, AH and MH, purchased from Showha Denko, Japan, were used as flame retardants.

### Preparation of the thermoplasticized crosslinked polyethylenes

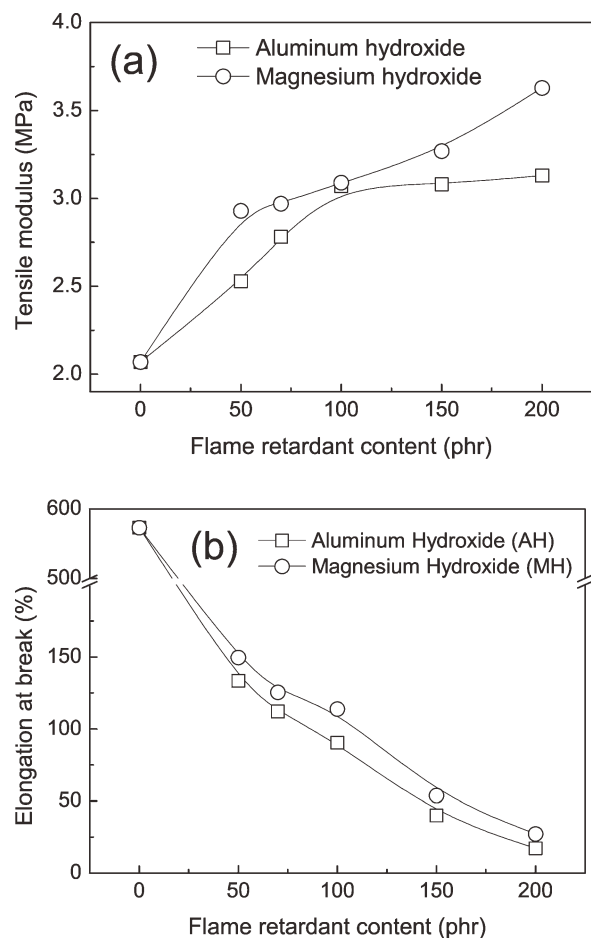
The thermoplasticized crosslinked PE were prepared using a supercritical methanol condition as reported in our previous article.<sup>4–6</sup> The supercritical decrosslinking reaction of crosslinked high-density PE with 88% gel fraction, was performed in a batch reactor. A batch reactor made of stainless steel (SUS316), described in a previous article was designed to perform the decrosslinking of crosslinked PEs under supercritical methanol. The reaction was carried out as follows: a 50 g piece of crosslinked PE and 100 mL of methanol were charged in a 500 mL stainless steel reactor. A piece of crosslinked PE with a size of 1–5 mm was prepared by chopping of a crosslinked PE pipe. Nitrogen was purged into the reactor to degas oxygen at room temperature. The reactor was heated to 370°C to start to decrosslink, kept for 5 min at 360°C, and then cooled to room temperature by cold water to terminate the reaction. The pressure at the reaction temperature was far above the methanol supercritical point of 240°C and 7.93 MPa. After the supercritical reaction, the products were filtered and dried in a vacuum oven. The thermoplasticized product had a residual gel fraction less than 10%.<sup>6</sup>

### Preparation of the PE/POE/metal hydroxides/LM composites

Two types of composites, PE/POE/AH/LM and PE/POE/MH/LM composites were prepared using

**TABLE I**  
Material Classifications in the UL94 Vertical Test According to ASTM D3801

Criteria conditions	V-0	V-1	V-2
Afterflame time for each individual specimen, $t_1, t_2$	≤10 s	≤30 s	≤30 s
Total after flame time, $t_1 + t_2$	≤50 s	≤250 s	≤250 s
Afterflame time plus afterglow time after second flame application, $t_2 + t_3$	≤30 s	≤60 s	≤0 s
Cotton indicator ignited by flaming particles or drops	No	No	Yes



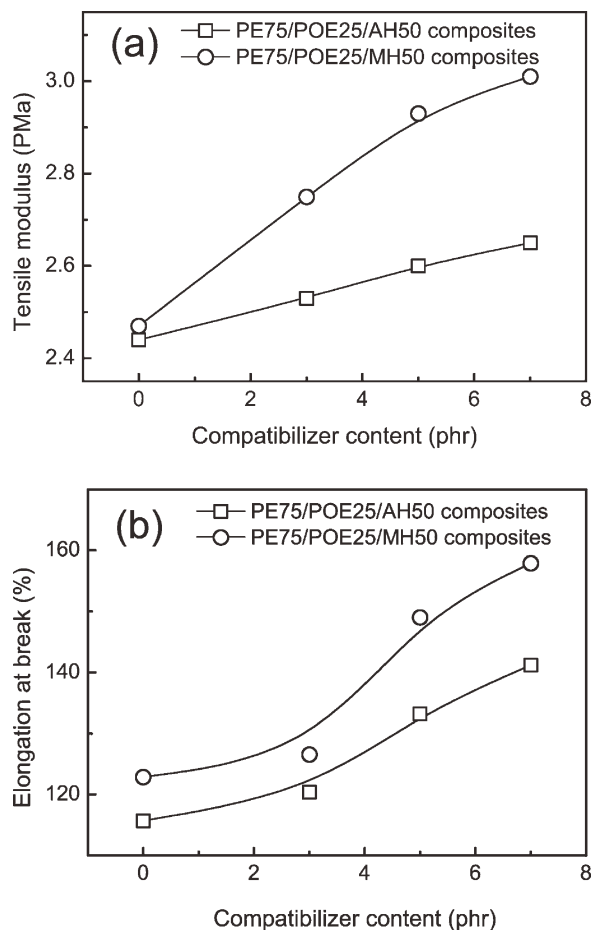
**Figure 2** (a) Tensile modulus and (b) elongation at break of the PE/POE/AH/LM and PE/POE/MH/LM composites at various flame retardant concentrations.

a twin screw extruder (PRISM TSE16TC, Thermo Electron) with a 16 mm barrel diameter and L/D = 15 at 210°C, respectively. POE was added to enhance the toughness of the composites. Before extrusion, the mixture of PE, POE, metal hydroxides, and LM was physically premixed and then extruded using the twin screw extruder at 210°C. The extrudate was dried in a vacuum oven. The specimens for determination of the mechanical properties and for non-flammability tests were prepared using a BOY 12A (BOY, Germany) injection molder.

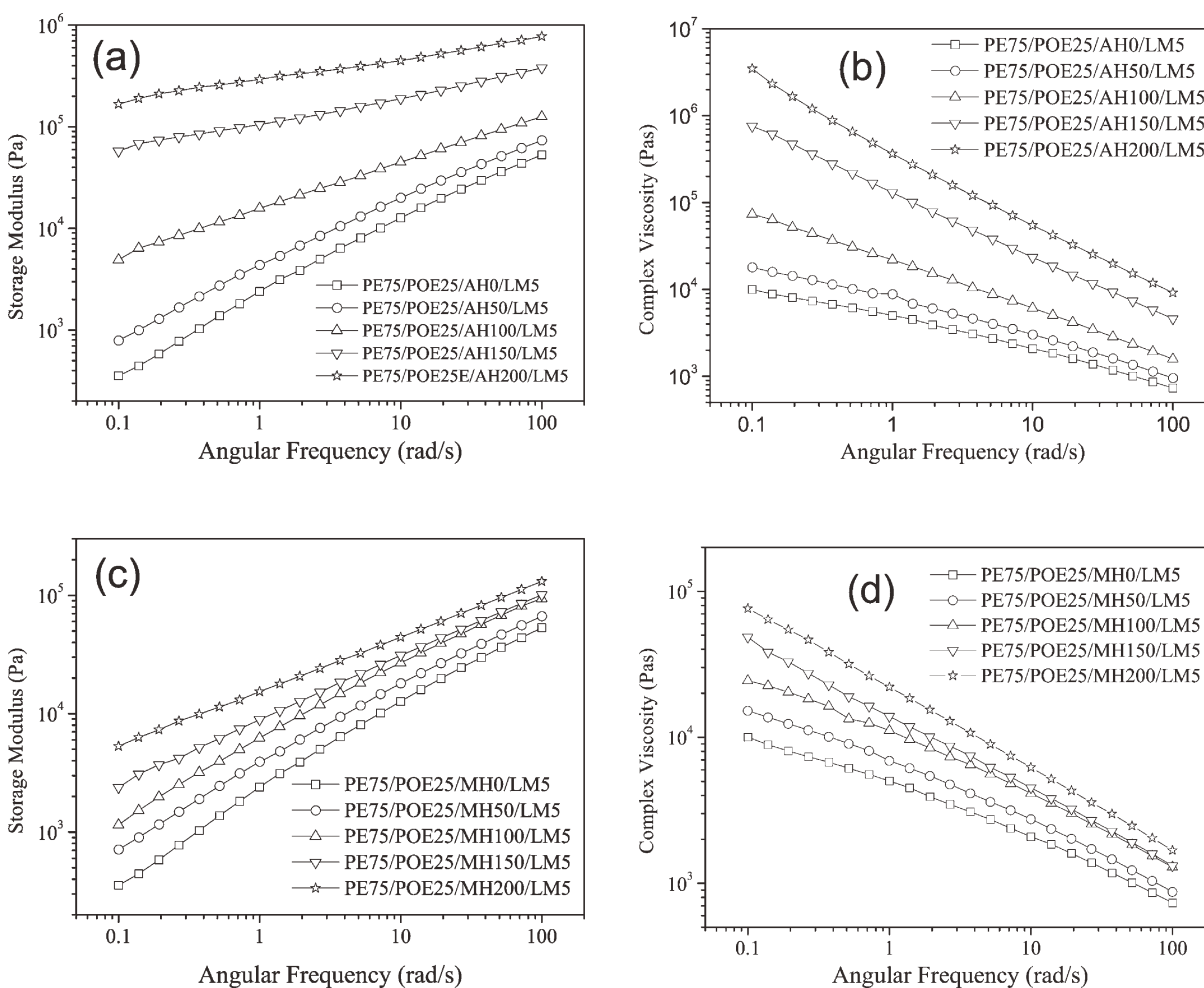
### Characterization

Tensile measurements were performed using an Instron UTM5567 according to ASTM D638. The dog-bone shaped specimens had a gauge length of 12 mm. Extension rate was kept at 50 mm/min. Tensile data were taken from the average value of 10 specimens. Rheological properties were measured using a UDS200 (Universal Dynamic Spectrometer, Physica, Austria). In rheological measurements, parallel plate geometry was adopted. The diameter and

gap of the parallel plate were 25 and 2.0 mm, respectively. All rheological measurements were performed in the linear viscoelastic region under nitrogen atmosphere. Morphological analysis was carried out using scanning electron microscopy (SEM) with a Hitachi S-2200C. Nonflammability tests were carried out using the limit oxygen index (LOI) and UL94V test methods. The sample dimension was 125 mm in length, 13 mm in width, and 3 mm in thickness. LOI measurements were performed using an ON-10, SUGA Test Instruments, according to ASTM D2863. The sample was held vertically in the glass column chamber, in which there was a controlled flow of oxygen and nitrogen mixture gas. The top of the sample was ignited to burn the sample. The test was repeated under various oxygen and nitrogen concentrations to determine the minimum concentration of oxygen LOI needed for burning of the sample. UL94 vertical burn tests were measured in accordance with ASTM D3801. The specimen was vertically fixed at the clip of the stand illustrated in Figure 1. Cotton was used as an indicator for the flaming particles or drops, and was



**Figure 3** (a) Tensile modulus and (b) elongation at break of the PE/POE/AH/LM and PE/POE/MH/LM composites at various LM compatibilizer concentrations.



**Figure 4** (a) Storage moduli and (b) complex viscosities of PE/POE/AH/LM composites, and (c) storage moduli and (d) complex viscosities of PE/POE/MH/LM composites at various flame retardant concentrations.

placed 300 mm below the specimen. A burner was placed 20 mm below the specimen. The burner produced a flame 20 mm high. The sample was ignited for 10 s using the flame from the burner. After flame time was measured, representing the length of time for which specimen continues to flame after the ignition source has been removed. The ignition was repeated two times.  $t_1$ ,  $t_2$  represent the after flame times for a first flame application and for a second flame application, respectively.  $t_3$  represents the after glow time, which is the length of time for which material continues to glow after the second flame application has been removed. The nonflammability criteria of V-0, V-1, and V2 were defined as listed in Table I.

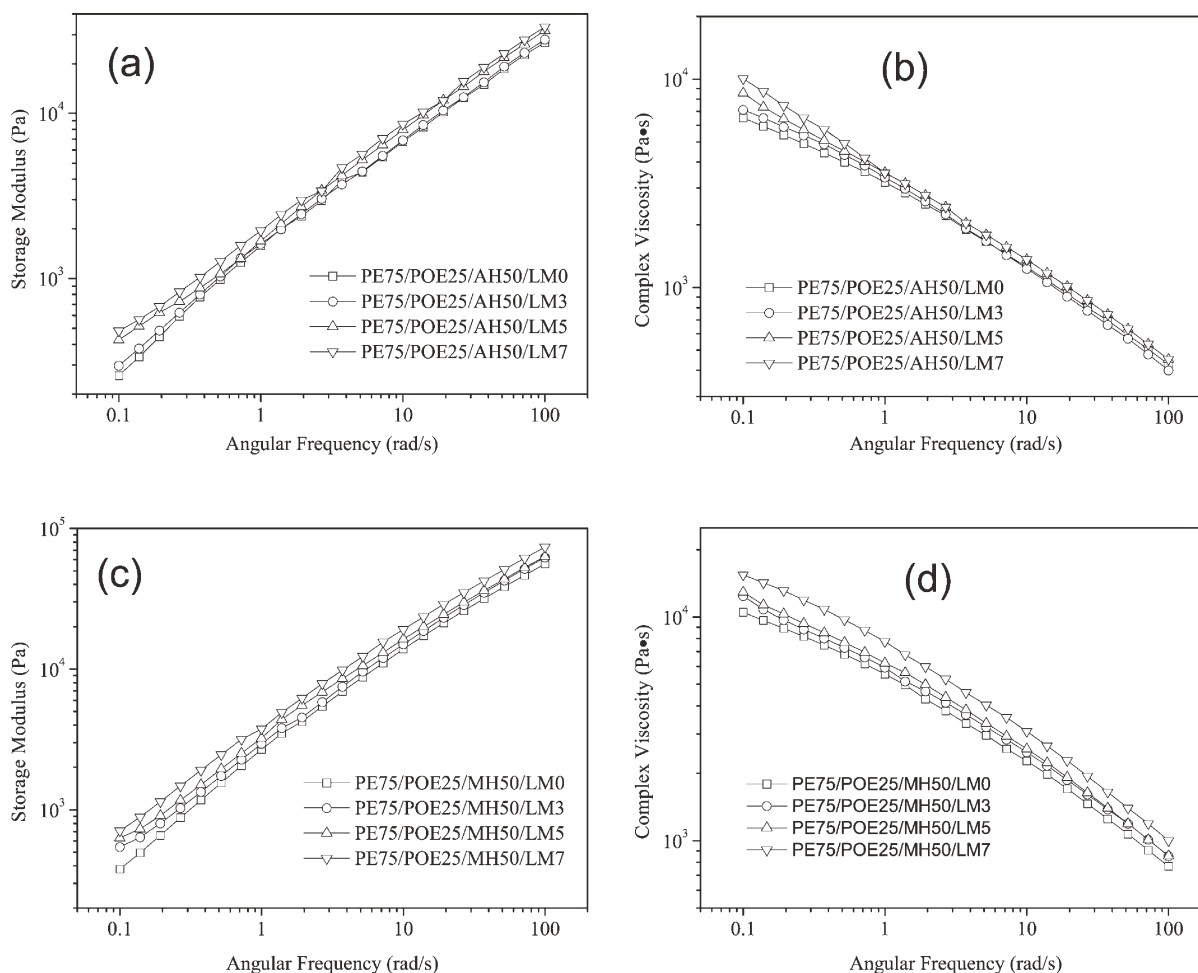
## RESULTS AND DISCUSSIONS

Figure 2 shows the tensile properties of the PE/POE/metal hydroxides/LM composites at various metal hydroxide flame retardant concentrations. The compositions of PE, POE, and LM were fixed at 75, 25, and 5 phr, respectively. The measure phr indi-

cates parts per hundreds resin. The matrix resin in this work consisted of the thermoplasticized cross-linked PE and POE mixture. As the concentration of the metal hydroxide flame retardant increased, the tensile modulus increased, while elongation at break decreased. The PE/POE/MH/LM composites with MH had higher tensile modulus and larger elongation at break than the PE/POE/AH/LM composites with AH at the same flame retardant concentration.

Figure 3 show the tensile properties of the PE/POE/metal hydroxides/LM composites at various LM compatibilizer concentrations. The compositions of PE, POE, and flame retardant were fixed at 75, 25, and 50 phr, respectively. As the concentration of the LM compatibilizer increased, not only the tensile modulus but also the elongation at break increased. The PE/POE/MH/LM composites with MH had higher tensile modulus and larger elongation at break than the PE/POE/AH/LM composites with AH at the same compatibilizer concentration.

Figure 4 shows the storage modulus  $G'$  and complex viscosity  $\eta^*$  of the PE/POE/metal hydroxides/LM

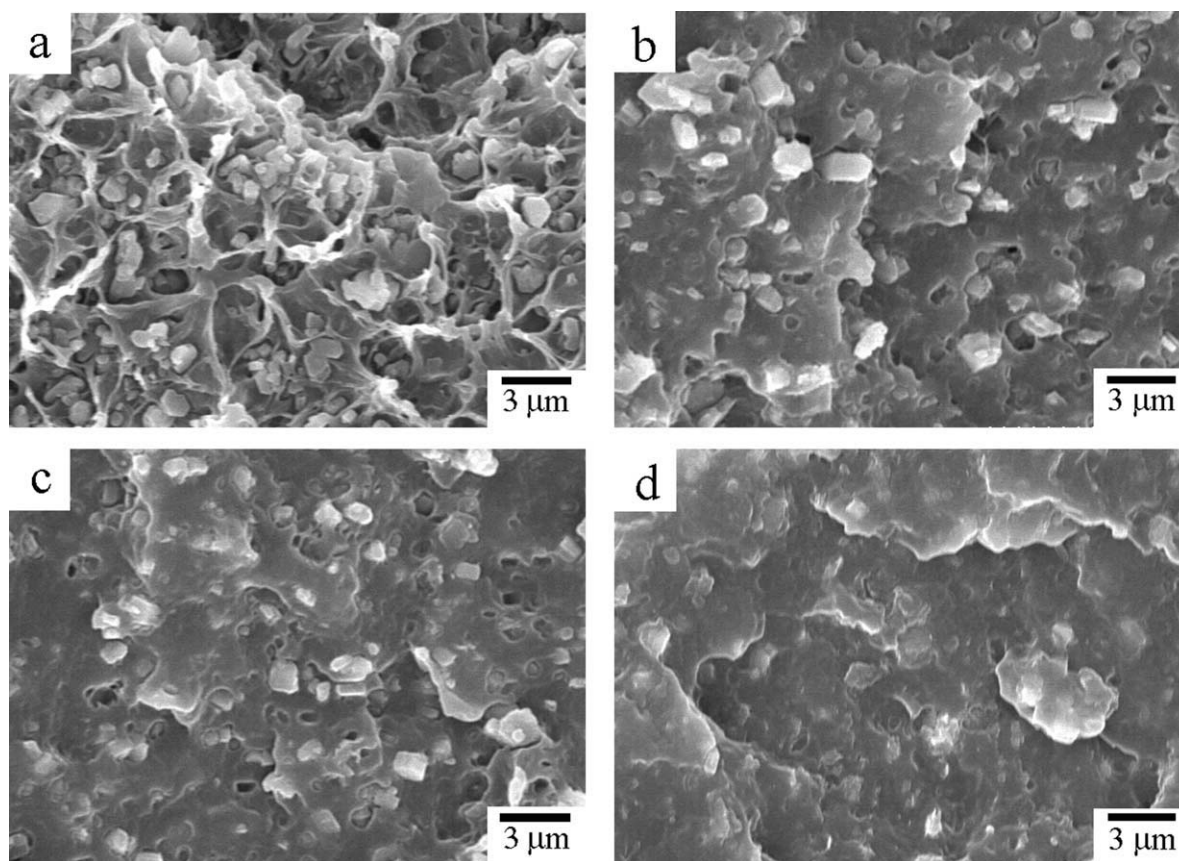


**Figure 5** (a) Storage moduli and (b) complex viscosities of PE/POE/AH/LM composites, and (c) storage moduli and (d) complex viscosities of PE/POE/MH/LM composites at various LM compatibilizer concentrations.

composites at various flame retardant concentrations. The compositions of PE, POE, and LM were fixed at 75, 25, and 5 phr, respectively. As the concentration of the metal hydroxide flame retardant in both PE/POE/AH/LM and PE/POE/MH/LM composites increased, both  $G'$  and  $\eta^*$  increased. The increases in the  $G'$  and  $\eta^*$  were much larger at low frequency than at high frequency. The frequency dependence of  $G'$  and  $\eta^*$  in the PE/POE/metal hydroxides/LM composites stems from the difference in the terminal slope of  $G'$  and  $\eta^*$  of the samples. Generally, in a liquid-like low-frequency region (known as a terminal region), the power law linear viscoelastic slopes of a monodisperse flexible homopolymer can be expressed as  $G'' \propto \omega^2$  and  $\eta^* \propto \omega^0$ . These slopes are very sensitive to changes in the chain branching,<sup>12</sup> crosslinking,<sup>13</sup> mesostructure of the polymers,<sup>14</sup> and filler incorporation.<sup>15,16</sup> In this work, the change of frequency dependence was from the flame retardant filler incorporation. As the flame retardant concentration increased, the filler-filler interaction increased because the inter filler distance decreased with the filler concentration.<sup>15,16</sup>

As a result, the terminal slope of  $G'$  decreased, while the terminal slope of  $\eta^*$  increased. At a high filler concentration, the composites behave like a quasi-solid that has the zero slope of  $G'$ . The PE/POE/AH/LM composites with AH had the same filler concentration dependency on the storage modulus and complex viscosity as the PE/POE/MH/LM composites with MH. The PE/POE/AH/LM composites had larger storage modulus and larger complex viscosity than the PE/POE/MH/LM composites. The PE/POE/AH/LM composites also had faster terminal slope decrease rate of  $G'$  than the PE/POE/MH/LM composites.

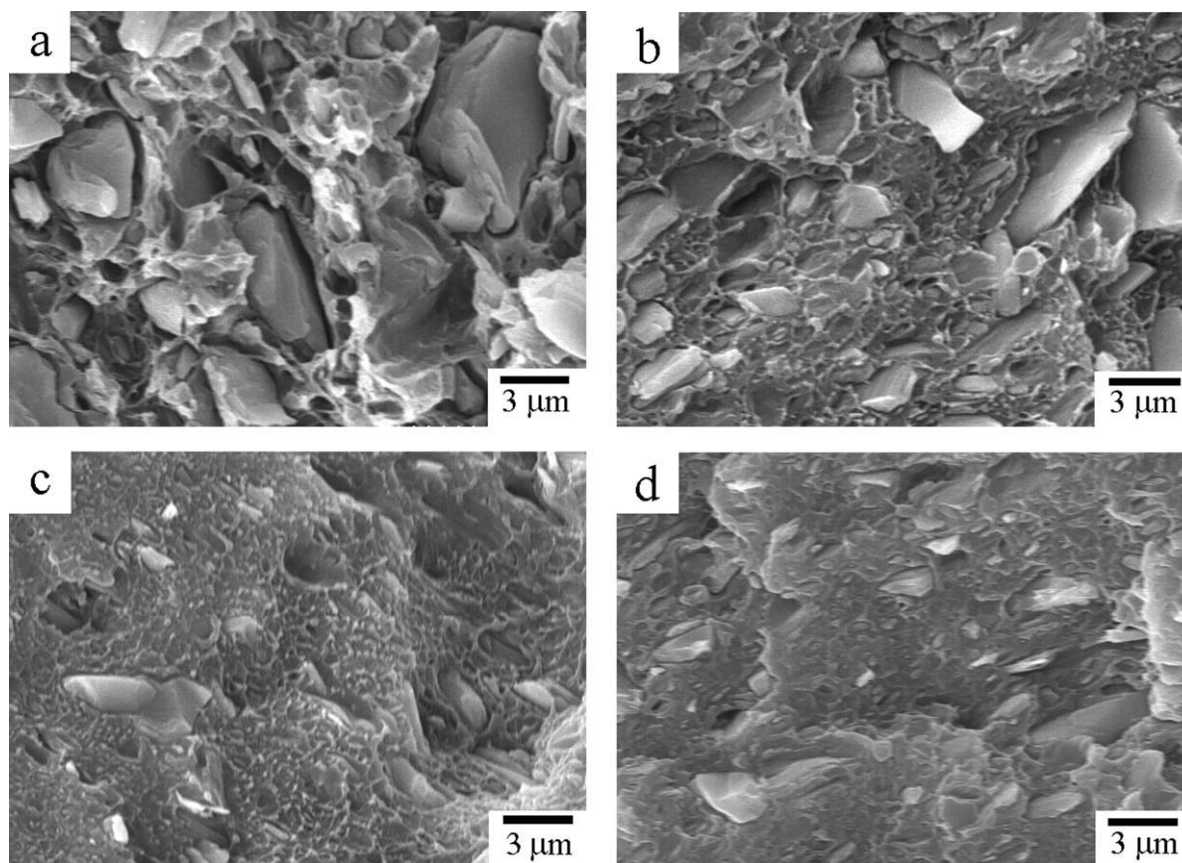
Figure 5 shows the storage modulus  $G'$  and complex viscosity  $\eta^*$  of the PE/POE/metal hydroxides/LM composites at various compatibilizer concentrations. The compositions of PE, POE, and flame retardant were fixed at 75, 25, and 50 phr, respectively. As the compatibilizer concentration increased, in both PE/POE/AH/LM and PE/POE/MH/LM composites,  $G'$  and  $\eta^*$  increased, and the terminal slope of  $G'$  decreased, while the terminal slope of  $\eta^*$  increased.



**Figure 6** SEM images of PE75/POE25/AH50/LM composites at various LM compatibilizer concentrations: (a) 0 phr, (b) 3 phr, (c) 5 phr, and (d) 7 phr.

Figures 6 and 7 show the SEM images of the PE/POE/AH/LM and PE/POE/MH/LM composites at various compatibilizer concentrations, respectively. In the PE/POE/AH/LM composites, the AH had an average particle size of 1  $\mu\text{m}$  and a narrow size distribution. For the PE75/POE25/AH50/LM0 composites without a compatibilizer, not only lot of aggregations consisting of several AH particles but also clear debonding between AH fillers and PE/POE matrix were observed. As the concentration of the LM compatibilizer increased, the dispersion state of the AH particles and adhesion between the particles and PE/POE matrix were improved. For the PE75/POE25/AH50/LM7 composites, the AH particles are well distributed in the matrix and the debonding between AH fillers and the PE/POE matrix did not appear. In the PE/POE/MH/LM composites, the MH had an average particle size of 3  $\mu\text{m}$  and quite broad sized distribution. The PE/POE/MH/LM composites show the same morphology dependency on the compatibilizer concentration as the PE/POE/AH/LM composites. As the compatibilizer concentration increased, the dispersion state of the MH particles and adhesion between MH fillers and PE/POE matrix were enhanced.

Figure 8a shows the LOI of PE/POE/metal hydroxides/LM composites at various flame retardant concentrations. The compositions of PE, POE, and LM were fixed at 75, 25, and 5 phr, respectively. The LOI is defined as a maximum percent concentration of oxygen in an oxygen and nitrogen mixture gas at which the object does not burn. A larger LOI value represents better nonflammability. As the concentration of the metal hydroxide flame retardant increased, LOI linearly increased. The PE/POE/MH/LM composites had larger LOI than the PE/POE/AH/LM composites at the same flame retardant concentration. Figure 8(b) shows the LOI of PE/POE/metal hydroxides/LM composites at various compatibilizer concentrations. The metal hydroxide content was fixed at 150, respectively. Interestingly, LOI of both composites depended on the concentration of the LM compatibilizer. LOI linearly increased with the concentration of the LM compatibilizer at the low compatibilizer concentration and then showed the maximum. A further increase of the compatibilizer concentration caused a decrease in the LOI. The PE/POE/MH/LM composites had larger LOI than the PE/POE/AH/LM composites at the same flame retardant concentration.

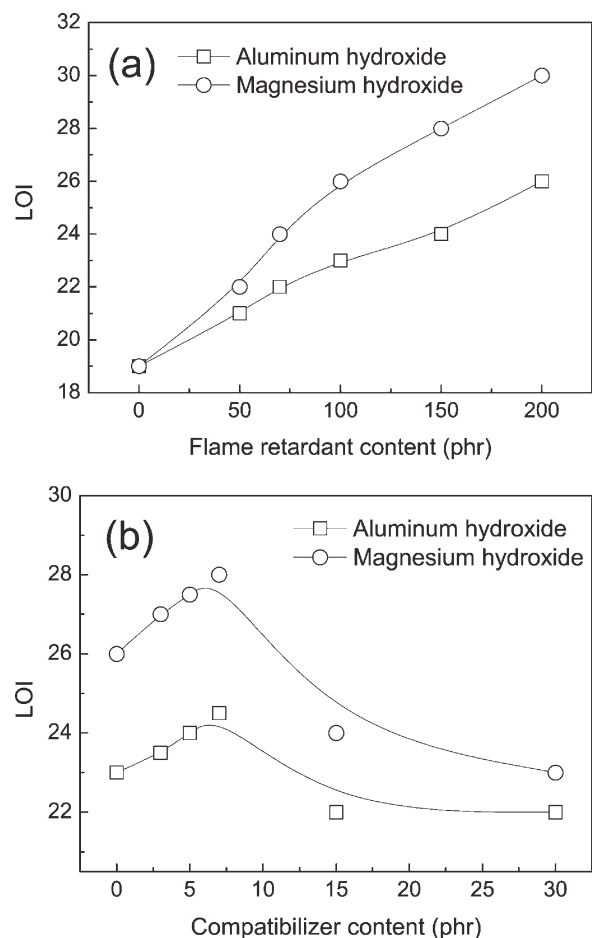


**Figure 7** SEM images of PE75/POE25/MH50/LM composites at various LM compatibilizer concentrations: (a) 0 phr, (b) 3 phr, (c) 5 phr, and (d) 7 phr.

Figure 9 shows the photographs of the PE/POE/AH/LM and PE/POE/MH/LM composites with various compatibilizer concentrations after the UL94 tests. The detailed UL94 vertical burning test results of the PE/POE/AH/LM and PE/POE/MH/LM composites are listed in Tables I and II. Burn means that the sample did not satisfy any UL94 test criteria of V-0, V-1, and V-2. The PE/POE/AH/LM composites were highly flammable, and showed the objects burning with soot, smoke and dripping, regardless of the flame retardant concentration or compatibilizer concentration. The nonflammability of the PE/POE/MH/LM composites depended not only on the flame retardant concentration but also on the compatibilizer concentration. As the MH flame retardant concentration increased, the nonflammability of the PE/POE/MH/LM composites was enhanced. The PE75/POE25/MH0/LM5 without MH flame retardant was burnt. But the PE75/POE25MH100/LM5 with 100 phr MH flame retardant had V2 UL94 index, and the PE75/POE25MH150/LM5 with 150 phr MH flame retardant had V0 UL94 index. The nonflammability of the PE/POE/MH/LM composites according to UL94 burning tests also depended on the concentration of the LM compatibilizer. The nonflammability was enhanced with the concentra-

tion of the LM compatibilizer at certain point and then deteriorated with the further increase in the compatibilizer concentration. The PE75/POE25/MH150/LM0 without the LM compatibilizer had V2 UL94 index, the PE75/POE25MH150/LM5 with the 7 phr LM compatibilizer had V0 UL94 index, while the PE75/POE25MH150/LM30 with the 30 phr LM compatibilizer was burnt, as shown in Figure 9(b).

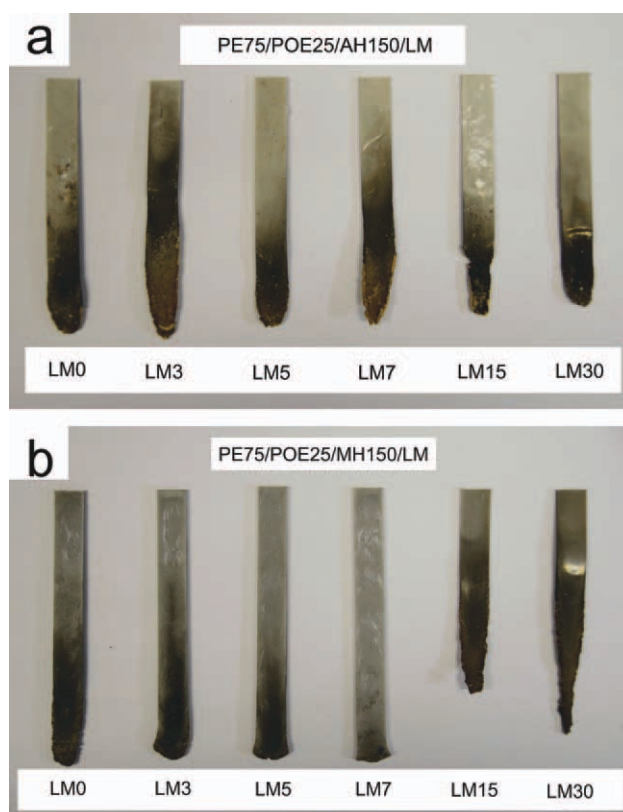
In this work, the thermoplasticized crosslinked PE and POE were used as a matrix polymer. The PE was decrosslinked from the crosslinked high-density PE using supercritical methanol. POE was used to enhance the toughness of the composites. Two types of metal hydroxides AH and MH and a low-density polyethylene-*g*-maleic anhydride (LM) were used as flame retardants and a compatibilizer, respectively. The PE/POE/AH/LM and PE/POE/MH/LM composites were successfully blended using a twin crew extruder. As the flame retardant concentration increased, in both composites, the tensile modulus increased, while elongation at break decreased. For the rheological measurements, as the flame retardant concentration increased, storage modulus  $G'$  of the composites increased, while the terminal slope of  $G'$  decreased. This can be simply understood by the fact that filler–filler interaction was enhanced with



**Figure 8** (a) LOI of the PE75/POE25/AH/LM5 and PE75/POE25/MH/LM5 composites at various flame retardant concentrations, and (b) LOI of the PE75/POE25/AH150/LM and PE75/POE25/MH150/LM composites at various LM compatibilizer concentrations.

the flame retardant filler concentration because the inter filler distance decreased with the filler concentration. The enhancement of the filler-filler interaction improved the mechanical stiffness (increase in tensile modulus), mechanical brittleness (decrease in elongation at break) and rheological pseudosolid-like behavior (decrease in terminal slope of  $G'$ ).

Similarly, as the compatibilizer concentration increased, in both PE/POE/AH/LM and PE/POE/



**Figure 9** (a) Photographs of PE75/POE25/AH150/LM and (b) PE75/POE25/MH150/LM composites at various LM compatibilizer concentrations after UL94 vertical burning tests. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

MH/LM composites, both tensile modulus and elongation at break increased. For the rheological measurements, storage modulus  $G'$  increased, while the terminal slope of  $G'$  decreased. As the compatibilizer concentration increased, not only the dispersibility of the flame retardants in the polymer matrix but also the adhesion between the flame retardants and the matrix polymer in both composites was improved, as shown in Figures 6 and 7, because the maleic anhydride groups in the LM can interact readily with functional groups on the inorganic flame retardant fillers, and long polymer chain in

**TABLE II**  
UL94 Nonflammability Test Results of PE/POE/AH/LM Composites

Fire retardant		Compatibilizer (phr)					
		LM 0	LM 3	LM 5	LM 7	LM 15	LM 30
AH (Al(OH) <sub>3</sub> )	0 phr	Burn	Burn	Burn	Burn	–	–
	50 phr	Burn	Burn	Burn	Burn	–	–
	70 phr	Burn	Burn	Burn	Burn	–	–
	100 phr	Burn	Burn	Burn	Burn	–	–
	150 phr	Burn	Burn	Burn	Burn	Burn	Burn
	200 phr	Burn	Burn	Burn	Burn	–	–



**TABLE III**  
**UL94 Nonflammability Test Results of PE/POE/MH/LM Composites**

Fire retardant		Compatibilizer (phr)					
		LM0	LM3	LM5	LM7	LM15	LM30
MH (Mg(OH) <sub>2</sub> )	0 phr	Burn	Burn	Burn	Burn	–	–
	50 phr	Burn	Burn	Burn	Burn	–	–
	70 phr	Burn	Burn	Burn	Burn	–	–
	100 phr	Burn	Burn	V2	V2	–	–
	150 phr	V2	V2	V0	V0	Burn	Burn
	200 phr	–	–	–	–	–	–

the LM are able to anchor to the PE/POE polymer matrix through physical entanglements and van der Waals interactions.<sup>10</sup> Therefore, the reinforcement effect of the flame retardant fillers was enhanced with the compatibilizer concentration.

From the nonflammability point of view, the nonflammability of both PE/POE/AH/LM and PE/POE/MH/LM composites was enhanced with the flame retardant concentration. The PE/POE/MH/LM composites with MH flame retardants had better nonflammability than the PE/POE/AH/LM composites with AH flame retardants at the same flame retardant concentration. It is well known that the MH flame retardants work as a better flame retardant for the polyolefin polymer than do the AH flame retardants.<sup>7,10,11</sup> The metal hydroxides release the water vapor to remove the heat away from the flame and reduce the formation of combustible gases. The char layer can be formed on the surface of the polyolefin polymer. The char layer insulates the polyolefin polymer from further thermal degradation. The decomposition temperature of MH is about 340°C and it is 200°C for AH. The higher decomposition temperature of MH makes the better flame retardancy for the polyolefin polymer because the decomposition temperature of MH is close to that of PE/POE matrix polymer.<sup>9</sup>

Interestingly, the nonflammability of the PE/POE/MH/LM composites was strongly affected by LM compatibilizer concentration. At low compatibilizer concentration, the nonflammability of the PE/POE/MH/LM composites was improved with the compatibilizer concentration. In contrast, at high compatibilizer concentration, the nonflammability of the PE/POE/MH/LM composites was deteriorated with the compatibilizer concentration. This demonstrates that small amount of the LM compatibilizer enhanced the nonflammability of the PE/POE/MH/LM composites owing to the fact that the compatibilizer improved not only the dispersibility of MH flame retardants in the PE/POE matrix but also the adhesion between the flame retardants and the matrix polymer in the composites. However, excess LM compatibilizer reduced the nonflammability of the composites due to the poor nonflammability of

the compatibilizer. As a result, this demonstrates that optimum concentration of the LM compatibilizer helps to enhance the efficiency of the MH flame retardants in the PE/POE/MH/LM polyolefin composites.

## CONCLUSIONS

Physical properties and nonflammability of the thermoplasticized crosslinked PE/POE composites with metal hydroxide flame retardants and a compatibilizer were investigated. The PE/POE/MH/LM composites with MH flame retardants had better nonflammability than the PE/POE/AH/LM composites with AH flame retardants at the same flame retardant concentration. Interestingly, the nonflammability of the polyolefin composites is strongly influenced by the compatibilizer concentration. Small amount of the compatibilizer enhanced the nonflammability of the PE/POE/MH/LM composites, while excess compatibilizer reduced the nonflammability of the composites. This demonstrates that optimum concentration of compatibilizer helps to enhance the efficiency of the MH flame retardants without a sacrifice of the mechanical brittleness in the polyolefin composites, and the thermoplasticized PEs using supercritical methanol process can be reused as nonflammable polyolefin composites for the cable and wire applications.

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