



The multilayered distribution of intumescent flame retardants and its influence on the fire and mechanical properties of polypropylene



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ABSTRACT

The multilayered distribution of intumescent flame retardants (IFRs) was obtained by fabricating a series of alternating multilayered composites consisting of polypropylene (PP) and IFR-filled PP (PPFR) layers through a layer-multiplying coextrusion technology. With increasing the layer numbers, the limiting oxygen index (LOI) and elongation at break (ϵ_b) of the multilayered composites were enhanced, accompanied with the gradually decreased values of the peak heat release rate and the total heat release. When the layer numbers beyond 16, the LOI and ϵ_b values were even larger than those of the PP/IFR conventional composite filled with the same content of IFRs. The layer interfaces and confined layer spaces in the multilayered system were considered to play a crucial role in retarding the spread of fire as well as the propagation of crazes. The significance of this work was providing an optimal route to fabricate flame-retarding composites with excellent mechanical properties.

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1. Introduction

The addition of intumescent flame retardants (IFRs) has been regarded as one of the most environment-friendly and promising methods to reduce the flammability of polymeric materials. During combustion, IFRs tend to decompose into expanded carbonaceous chars, acting as an insulating barrier to reduce heat and fuel transfer between the heat source and the polymer and limit the diffusion of oxygen into the material. Hence, in order that enough char contents can be generated to inhibit the spread of fire, a large amount of filler loading is generally required which in turn causes deteriorated mechanical properties and processing issues [1,2].

Actually, it is recognized that the content of IFRs satisfying the flame-retarding requirement of the polymeric matrix is dependent on both the chemical structure and the distribution of fillers [3]. Structural modification is regarded as a direct and effective way to enhance the flame-retarding efficiency of IFRs. However, as the IFR type is chosen, the distribution would be a key factor in order to achieve the maximum performance of the whole composite system [4,5].

In most cases, IFRs are randomly distributed in polymeric matrix. A large reduction in flammability would be approached accompanied with the formation of a network-structured protective layer during burning [6]. Hence, there should be a critical distance (D_C) between IFR particles. Only when the distance is less than D_C , the expanded carbonaceous chars would contact each other acting as a flame-retarding layer. It can be imagined that

there are two kinds of basic ways for IFR distribution: uniform and multilayered distribution as schematically displayed in Fig. 1.

A uniform distribution state is commonly pursued [7–10]. For example, Pack et al. [7] and Song et al. [8] improved the dispersion of IFRs by increasing their compatibility with polymeric matrix. Wang et al. [9] and Isitman et al. [10] introduced inorganic fillers (such as clays, fibers and nanotubes) into an IFR-filled polymeric system for breaking the aggregation of IFRs and making them uniformly dispersed in the matrix. Combustion results displayed that a uniform IFR distribution was indeed beneficial to the formation of even and compact char layers. However, it is recognized that the random distribution of IFRs would also promote the propagation of crazes (or cracks) in the matrix which may result in the deterioration of mechanical toughness.

Compared to the uniform distribution, the multilayered distribution of IFRs is less reported and mainly applied in the layer-by-layer deposition system. Laufer et al. [11], Li et al. [12], and Carosio et al. [13] found that the multilayered coating effectively prolonged the time to ignition and reduced the heat release rate of polymeric substrates. However, few studies related to the influence of the multilayered distributing state of IFRs on the flame-retarding properties of the matrix are concerned, to the authors' best knowledge. Besides, Sung et al. [14,15] studied the mechanical behaviors of alternating multilayered materials consisting of polycarbonate (PC) and styrene–acrylonitrile copolymer (SAN) layers. It was found that the crazes originated from SAN layers could be terminated by shear bands in adjacent PC layers through interfacial adhesion, leading to the increase of the strength and toughness of the whole material. Hence, it is imagined that the multilayered distribution of

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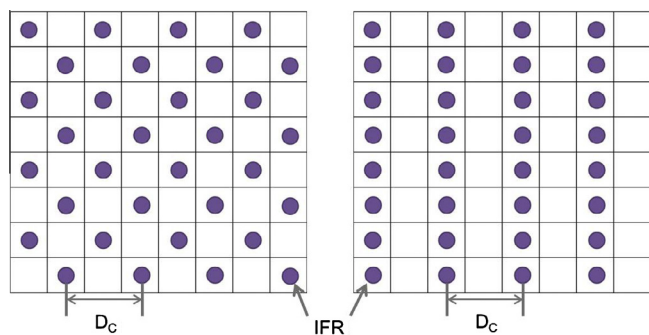


Fig. 1. Schematic of the uniform (left) and multilayered (right) distribution of IFRs in the matrix.

IFRs may provide one of optimal routes to fabricate flame-retarding composites with excellent mechanical properties.

In this work, the multilayered distribution of IFRs was obtained by preparing a series of alternating multilayered composites consisting of pure polypropylene (PP) and IFR-filled polypropylene (PPFR) layers through a layer-multiplying coextrusion technology. The distributing state of IFRs was controlled by adjusting the number of layers or the thickness ratio of PPFR and PP layers. For comparison, the conventional composites with a uniform distribution of IFRs were also prepared by filling with the same content of IFRs as that of the corresponding multilayered system. The effects of the multilayered and uniform distribution of IFRs on the fire and mechanical properties of the PP matrix were compared and investigated schematically.

2. Experimental

2.1. Materials

PP (T30S), with a density of 0.899 g/cm^3 and a melt index of 3 g/10 min, was provided by Lan Zhou Petroleum Chemical Company Ltd. (China). Commercial IFRs (6000-1), with a mean size of about $1 \mu\text{m}$, were supplied by Dongguan Dohar Chemical Company Ltd. (China).

2.2. Specimen preparation

Prior to the layer-multiplying coextrusion, PP/IFR pellets (denoted as PPFR) filled with 37.5 wt% IFRs were prepared using a twin-screw extruder. Subsequently, the PPFR/PP alternating multilayered composites were prepared using the layer-multiplying coextrusion system, the mechanism of which was described previously [16]. As illustrated in Fig. 2(a), the dried PPFR pellets and pure PP were extruded from different extruders (A and B) forming a 2-layer melt in the coextrusion block (D) and then went through an assembly of layer-multiplying elements (LMEs, E). The PPFR/PP alternating multilayered composites with 2, 4, 8, 16 and 32 layers were prepared by using 0, 1, 2, 3, and 4 LMEs, respectively. The thickness ratios of PPFR and PP layers (6.5:9.5, 8:8, and 9.5:6.5) were obtained by controlling the extruding rate of each extruder. The corresponding relationship between the content of IFRs in the whole multilayered system and the thickness ratio of PPFR and PP layers is shown in Fig. 2(b). It should be noted that the content of IFRs in PPFR layers was maintained at 37.5 wt%, irrespective of the number of layers and the thickness ratio of the PPFR and PP layers. The number of layers, the overall thickness, the extruding rate and the PP layer thickness of the PPFR/PP multilayered composites are summarized in Table 1.

For comparison, the PP/IFR conventional composites filled with 17.1, 20.7 and 24.1 wt% IFRs were prepared using only one extruder of the layer-multiplying system by applying 4 LMEs. In this paper, these PP/IFR composites were referred as single-layer samples where IFRs were dispersed evenly in PP matrix.

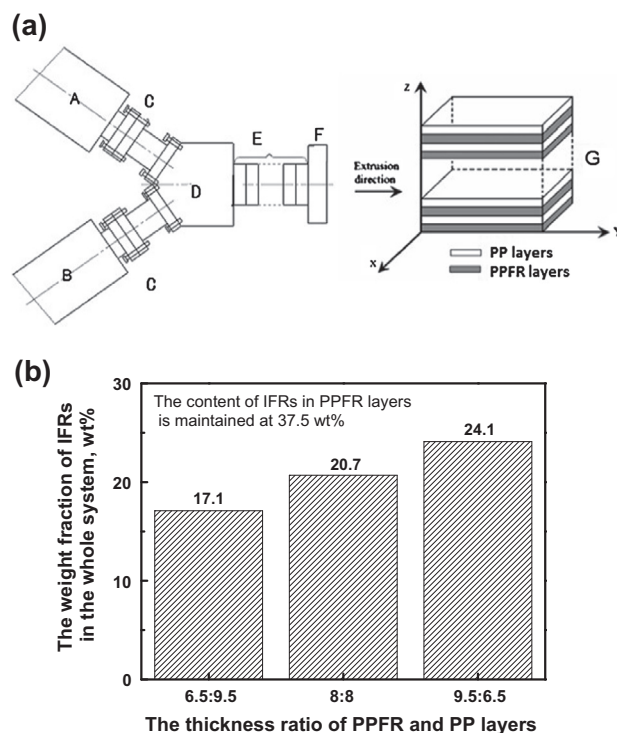


Fig. 2. (a) Schematic of layer-multiplying coextrusion system: A, B—extruders; C—connector; D—coextrusion block; E—layer multiplying elements; F—die; G—the specimen with an alternating multilayered structure. (b) The corresponding relationship between the content of IFRs in the whole multilayered system and the thickness ratio of PPFR and PP layers.

2.3. Characterization

The polar light microscope (PLM) observation was performed using a PLM (XSZ-H, Chongqing Photoelectric Instrument Co., China) equipped with a video camera. A thin slice about $10 \mu\text{m}$ in thickness was obtained by a microtome from each sample along the extrusion direction.

All thermogravimetric tests were performed on a thermogravimetric analyzer (TGA, TG-209F1, NETZSCH, Germany). Each sample, with a weight of 5–10 mg, was heated from 30 to $700 \text{ }^\circ\text{C}$ at a rate of $10 \text{ }^\circ\text{C/min}$ in air.

Fourier transform infrared (FTIR) spectra were taken on a Nicolet-IS10 (Thermo Electron Co., USA) spectrometer from 4000 to 650 cm^{-1} with a 4 cm^{-1} resolution. Absorption bands between 3700 and 800 cm^{-1} were analyzed.

The limiting oxygen index (LOI) values were measured by an oxygen index meter (HC-2C, Jiangning Analytical Instrument Factory, China). The dimensions of each specimen are $130 \times 6.5 \times 1.4 \text{ mm}^3$. An FTT cone calorimeter was used to evaluate the flammability of samples under an external heat flux of 35 kW/m^2 , with specimen dimensions of $100 \times 100 \times 2.8 \text{ mm}^3$.

Tensile tests were performed using an Instron 4302 tension machine (Canton, MA, USA) at a crosshead speed of 20 mm/min in accordance with ASTM D638. At least of five specimens for each sample were tested and the average value was calculated.

3. Result and discussion

3.1. Uniform and multilayered distribution of IFRs

The cross-sectional morphologies of PPFR/PP multilayered and PP/IFR conventional composites were observed through PLM. Micrographs in Fig. 3(a–c) display the multilayered distribution

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