

Synergistic effect of decabromodiphenyl ethane and montmorillonite on flame retardancy of polypropylene

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ABSTRACT

Polypropylene (PP) is melt-compounded in a twin-screw extruder with surface-modified decabromodiphenyl ethane/antimony trioxide (DBDPE/Sb₂O₃) and organically modified montmorillonite (OMMT). The intercalation and dispersion microstructure of OMMT in the nanocomposites are investigated by X-ray diffraction, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Thermal stability and char residue are characterized by thermogravimetric and differential thermal analysis (TGA–DTA). Flame retardant properties are evaluated by limited oxygen index (LOI) and UL-94 vertical burning test.

The results indicate that better flame retardancy can be achieved for the composite containing a modified mixture DBDPE/Sb₂O₃. The presence of DBDPE/Sb₂O₃ could improve the dispersion of OMMT in polypropylene, leading to higher thermal stability and more char residue. A synergistic effect between OMMT and DBDPE/Sb₂O₃ has been observed and discussed.

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1. Introduction

Polypropylene (PP) has been widely used in numerous products, such as electrical cables, architectures, packaging and transportation, due to its excellent mechanical properties. However, pure polypropylene is highly flammable at room temperature and leaves almost no char. Flame retardants are usually required to be added during compounding to improve the fire retardancy of PP. Many flame retardants have been developed for PP in the last three decades, for example, halogen- or phosphorus-containing compounds, silicon additives, intumescent flame retardants, metallic hydroxides and oxides, nanometer composites. Chlorinated and brominated organic compounds, such as polybrominated biphenyls (PBB), polybrominated diphenyl ethers (PBDE), tetrabromobisphenol A (TBBPA), and hexabromocyclododecane (HBCD) are still highly used all over the world, although they are encountering more pressure from environmental protection organizations [1,2]. PBB and PBDE have been the most popular flame retardants, because of their good flame retardant efficiency, excellent thermal

stability, high decomposition temperature (>350 °C), and relative low loading [3–7].

Since 1980s, materials with PBB (PBB) were found that their thermal breakdown products contained polybrominated dibenzofurans (PBDF) and polybrominated dibenzo-*p*-dioxins (PBDD), which had severely caused environmental concerns. It was reported that high levels of PBDF/PBDD were detected in marine mammals, aquatic wildlife, even in human breast milk and blood [8,9]. European Union (EU) directive on the “Reduction of certain hazardous substances in electrical and electronic equipment” (RoHS, 2002/95/EC) prohibited the use of PBB/PBBE, nevertheless, the European Commission lifted parts of the ban in July 2005, that PBB/PBBE could be used under commanded levels (below 1000 ppm) [10].

However, other brominated flame retardants (BFR) have been investigated to displace PBB/PBDE. Decabromodiphenyl ethane (DBDPE, Saytex 8010) was successfully developed by Albemarle Company (America), in the 1990s [5,11,12], which has the following attractive features:

- (1) Good thermal stability, suitable for high temperature processing of polymers.
- (2) Does not contain ether bond and will not generate PBDF/DD, which can be widely used without the limitation of RoHS.
- (3) Good anti-ultraviolet capacity and brightness stability, and free-transference.

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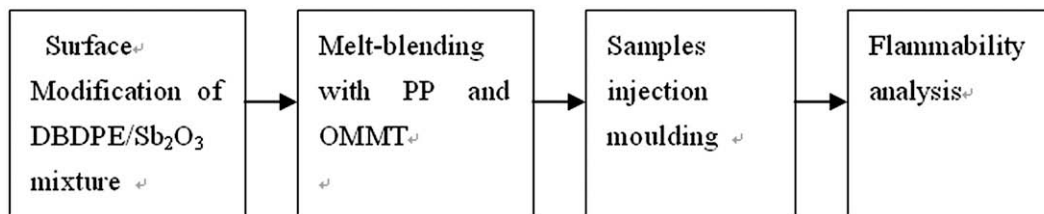


Fig. 1. Schematic process of the experiment.

- (4) Similar levels of bromine (containing more than 81.5% of Br) and molecular weight (about 971.0), with identical flame retardancy at low loading.
- (5) Be able to be recycled.

Traditionally, the halogenated organic compounds are usually used in incorporation with antimony trioxide compounds (Sb_2O_3) to enhance their flame retardancy (halogen–antimony synergistic) [13,14]. Many studies show that antimony trioxide can increase smoke production and has toxicity issues, so other compounds like zinc borates have been tried to substitute it. However, halogen–antimony synergistic system is still widely used due to their excellent fire resistance [15,16].

It has been shown that compounding of layered silicates with polymers is a new and promising way to enhance the flame retardancy. Positive impacts on mechanical, optical, thermal and electrical properties have been observed with clay loading of less than 10 wt%. The maximum rate of heat release could be reduced up to 70% [17,18]. Montmorillonite (MMT) is a typical layered clay mineral with a central alumina octahedral sheet sandwich between two silica tetrahedral sheets. Depending on compatibility/wettability of MMT with polymers, MMT can be intercalated, exfoliated, or just dispersed as aggregates in polymers. The intercalated and exfoliated nanocomposites usually show improved fire retardancy and mechanical properties, in which exfoliated system usually can bring better performances [19,20].

In this work, PP/DBDPE/ Sb_2O_3 /OMMT nanocomposites were prepared by melt-compounding, followed by the characterization of fire performance, thermal stability and char morphology. The synergistic effect of DBDPE/ Sb_2O_3 and OMMT on flame retardancy of PP has also been investigated.

2. Experimental

2.1. Materials

The polypropylene (PP) with the trade name of K1001 used was supplied by Yan Shan Petroleum (China).

The organically modified montmorillonite (OMMT) with dimethyl, dihydrogenated tallow (2M2HT) was obtained from Southern Clay Product Inc (USA), under a trade name of Cloisite® 15A.

Decabromodiphenyl ethane (DBDPE), with an average particle size of 2.5 μm , and around 81.5 wt% of bromine was purchased from Shangdong Runke Chemical Co. Ltd. (China).

Antimony trioxide (Sb_2O_3), ethanol, three silane coupling agents (γ -glycidylpropyltrimethoxy silane (GTMS), vinyltrimethoxy silane (VTMS), γ -aminopropyltrimethoxy silane (APS)) were all analytical grade, supplied by Tianjin Fuchen Chemical Reagent Factory (China).

2.2. Preparation of the nanocomposites

Flame retardant particles as DBDPE, Sb_2O_3 , OMMT and processing additives were pre-dried at 80 °C for 12 h in an oven with circulating air. Silane coupling agent was diluted with ethanol at the mass ratio of 1:1. Dried flame retardants of DBDPE and Sb_2O_3 with a ratio of 4:1 were physically mixed thoroughly (will be called DBDPE/ Sb_2O_3 mixture in the following discussion), and then surface-modified with silane coupling agent with different chemical structures and hydrophilic groups (such as γ -glycidylpropyltrimethoxy silane (GTMS), vinyltrimethoxy silane (VTMS) and γ -aminopropyltrimethoxy silane (APS), with the amount of 2 wt%

Table 1
Flammability properties of composites containing DBDPE/ Sb_2O_3 treated with different silane coupling agents.

Code	Silane coupling agents	Molecular structures	Boiling temperature (°C)	Content (wt%)	LOI value (%)	UL-94 rating
1	/		/	/	23.1	No-rating
2	GTMS	$\begin{array}{c} \text{OCH}_3 \\ \\ \text{CH}_2 - \text{CH} - \text{CH}_2 - \text{O} - (\text{CH}_2)_3 - \text{Si} - \text{OCH}_3 \\ / \quad \backslash \\ \text{O} \quad \quad \\ \quad \quad \quad \text{OCH}_3 \end{array}$	290	2	23.4	V-2
3	VTMS	$\begin{array}{c} \text{OCH}_3 \\ \\ \text{CH}_2 = \text{CH} - \text{Si} - \text{OCH}_3 \\ \\ \text{OCH}_3 \end{array}$	122	2	22.2	No-rating
4	APS	$\begin{array}{c} \text{OCH}_3 \\ \\ \text{NH}_2 - (\text{CH}_2)_3 - \text{Si} - \text{OCH}_3 \\ \\ \text{OCH}_3 \end{array}$	217	2	24.3	V-0

DBDPE: Sb_2O_3 = 4:1 (w/w), and their total weight percent is 30% based on PP.

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