Adsorption behaviors of iodide anion on silver loaded macroporous silicas (Post-print)

Authors: WU Hao, WU Yan, CHEN Zi, WEI Yue-Zhou

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Abstract

A macroporous silica-supported silver adsorbent was synthesized by grafting a silver thiourea complex (Ag(tu)3NO3) onto a silica copolymer support (SiO2-P). The adsorbent was employed for iodide ion (I-) adsorption using both batch and column techniques. Kinetic and saturation adsorption experiments were conducted by varying the shaking time and initial I- concentration. Experimental results showed that the kinetic adsorption process of I- follows a pseudo-second-order model, while the saturation adsorption follows a chemisorption mechanism and conforms to the Langmuir adsorption isotherm equation. The I- breakthrough curve exhibits an S-shaped profile. Column efficiency is estimated to exceed 90%.

Full Text

Preamble

Irradiation and Flame Retardant Effect of Poly[bis(phenoxyphosphazene)] and Magnesium Hydroxide in LDPE Composites

Jian-Xi Li[1][2], Cong Zhang[1], Tao Chen[1], Lin-Fan Li[1], and Jing-Ye Li[1]†

[1]CAS Center for Excellence on TMSR Energy System, Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai 201800, China
[2]University of Chinese Academy of Sciences, Beijing 100049, China

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Poly[bis(phenoxyphosphazene)] (PBPP) and magnesium hydroxide (MH) are used as a flame retardant blend with low-density polyethylene (LDPE) for nuclear cable applications. This study investigates the effects of PBPP in MH-LDPE blend composites on flame retardancy and electron beam irradiation resistance. The structure, morphology, and properties of the blend composites

irradiated by an electron beam to different absorbed doses were characterized. The results indicated that PBPP provides lubrication during processing. As the PBPP content in the blend increases at 20 phr MH, the material becomes easier to process, as evidenced by increased melt flow rate. Higher PBPP content correlates with higher limiting oxygen index values. The elongation at break of PBPP-containing composites (at 50 phr MH) was significantly higher than that of non-PBPP composites at different absorbed doses. Thermogravimetric analysis results suggested that the improved mechanical properties resulting from electron-beam irradiation could be attributed to the consumption of PBPP.

Keywords: Flame retardant, Electron beam irradiation, Low-density polyethylene, Poly[bis(phenoxyphosphazene)], Magnesium hydroxide
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Introduction

Low-density polyethylene (LDPE) plays an important role in the plastics industry due to its excellent properties, including good mechanical characteristics, chemical resistance, and ease of processing [1, 2]. However, LDPE is highly flammable. Extensive research has been conducted to improve its flame retardancy, generally through two approaches: the 'additive' type and the 'reactive' type [3]. Additive-type flame retardants are widely used in LDPE and fall into two main categories: (1) Inorganic fillers such as nano-clay, metal oxides, and metal hydroxides. However, high loading levels result in deteriorated mechanical properties due to poor compatibility with the polymer matrix. (2) Organic additives, most of which are halogen-containing flame retardants [4]. These are toxic to the environment and humans during combustion, creating demand for halogen-free alternatives.

Polymer combustion is influenced by five factors: primary elements, heat, oxygen, combustible materials, and radical reactions. The combustion process in air occurs in three stages: (1) thermal decomposition of the polymer producing flammable gases, (2) combustion of these products in air, and (3) heat from combustion promoting continued breakdown of solid or molten materials. Flame retardants function by interrupting one or more of these stages to prevent or slow combustion. Phosphorus-containing flame retardants are particularly versatile, undergoing reactions during combustion that transform phosphorus compounds into phosphoric acid, then metaphosphoric acid, and finally metaphosphoric acid copolymers [5]. The generated metaphosphoric acid copolymer forms a vitreous protective film that covers the burning zone and cuts off oxygen supply. Both phosphoric acid and metaphosphoric acid copolymer exhibit strong dehydration and carbonization properties. The resulting carbonized layer blocks oxygen diffusion and prevents combustion while its poor thermal conductivity slows heat transfer between the internal polymer and external heat source. Additionally, phosphorus-containing flame retardants generate $PO\cdot$ radicals that substantially absorb $H\cdot$ and $HO\cdot$ radicals, thereby interrupting combustion reactions. While nitrogen-containing flame retardants alone have limited efficacy, they are often used with phosphorus flame retardants because nitrogen compounds promote carbonization of phosphorus-containing compounds, creating a synergistic nitrogen-phosphorus effect [6].

Polyphosphazenes are inorganic-organic hybrid polymers in which inorganic elements are incorporated into the organic backbone chain. Poly[bis(phenoxyphosphazene)] (PBPP) is a promising flame retardant candidate [7-10]. The alternating double bonds (-N=P-N=P-) in the backbone create a π-π conjugated structure that can resist electron beam irradiation and protect the polymer matrix [11-15]. Additionally, each repeating unit contains two phenoxy groups, creating a P-N synergistic effect. These polymer composites can be introduced into materials for nuclear applications due to their excellent flame retardant properties, though specialized studies on nuclear power station cable applications remain limited in China [16, 17]. This work investigates PBPP-MH-LDPE composites for flame retardant performance and resistance to electron beam irradiation.

II. Experimental

A. Materials

LDPE with a melting point of 112 °C was supplied by UBE Corporation, Japan. MH (Magnifin H10) with a median particle size of 0.80–1.10 µm was obtained from Albemarle Corporation. PBPP with a polymerization degree of 2–3 was purchased from Zibo Blue Chemical Company Limited, China.

B. Composite Preparation

LDPE-based composites were mixed with MH and PBPP in phr (parts per hundred resin based on 100 parts of LDPE) using a double roller mill SK-160B (Shanghai Rubber Machinery Works, China) at 140 °C and 8 rpm for 10 minutes. After mixing, the composites were hot-pressed under 10 MPa into 1 mm thick sheets for 5 minutes at 160 °C. The composites are designated as PE–x–y, where x represents the phr value of MH and y represents the phr value of PBPP. Formulation details are listed in Table 1 .

C. Irradiation

Electron-beam irradiation was performed in air using a KFG-1 electron beam accelerator at 2.5 MeV acceleration voltage and 10 mA current. The dose rate was 15 kGy per pass, with total absorbed doses ranging from 0–1000 kGy.

D. Characterization

Fourier-transform infrared spectroscopy in attenuated total reflectance (FTIR-ATR) mode was used to study chemical alterations on the composite surface.

Measurements were performed on a Perkin Elmer Spectrum 100 FTIR spectrometer with 4 cm^{-1} resolution and 32 scans per spectrum from 4000 cm^{-1} to 500 cm^{-1}.

Surface morphology was examined using a JEOL JSM-6500F scanning electron microscope (SEM) at 10 kV acceleration voltage. Samples were fractured after cooling in liquid nitrogen and coated with a thin Au layer prior to examination.

Density was measured using a Toyoceike DH-100 automatic densitometer (Japan) according to ASTM D-1505.

Melt flow rate (MFR) tests were conducted according to ASTM D1238 using an SRZ-400E Melt Flow Tester (Changchun, China). Four automatically timed measurements were taken at 190 °C under a 2.16 kg load.

Tensile strength and elongation at break were measured using an Instron 5965 electronic tensile strength meter (UK) with a 500 N load cell, following ASTM D882-02 method at a crosshead speed of 250 mm/min and gauge length of 20 mm at room temperature. At least five samples per composite were tested to obtain reproducible average values.

Limiting oxygen index (LOI) values were determined using an HC-2 Oxygen Index instrument (Jiangning Analysis Instrument Factory, China) on 120 mm × 6 mm × 3 mm sample sheets according to ASTM D2863. UL-94 horizontal-vertical burning tests were performed according to GB/T2408-1996 on 130.0 mm × 13.0 mm × 1.6 mm samples using a CZF-3 apparatus (Cangzhou Ke Shuo Construction Equipment Ltd., China).

Thermogravimetric analysis (TGA) was conducted using a NETZSCH TG 209 F3 Tarsus Thermo-Microbalance (Germany) from room temperature to 700 °C at 10 °C/min heating rate under nitrogen atmosphere.

III. Results and Discussion

A. Chemical Structure Analysis

Figure 1 [Figure 1: see original paper] shows ATR-FTIR spectra of blend composites from 4000 cm^{-1} to 500 cm^{-1}. The MH-LDPE blend composite exhibited five main bands (Fig. 1c): 2918 cm^{-1} and 2849 cm^{-1} (CH_2 symmetrical and asymmetrical stretching), 1465 cm^{-1} (CH bending deformation), 720 cm^{-1} (CH rocking deformation), and 3694 cm^{-1} (-OH stretching vibration in MH crystals).

In PE-20-5 and PE-20-12.5 blend composites, bands at 1590 cm^{-1} and 1486 cm^{-1} corresponded to phenyl group framework vibrations, while peaks at 767 cm^{-1} and 685 cm^{-1} indicated mono-substituted benzene rings. Bands at 1263 cm^{-1} and 1176 cm^{-1} were attributed to P=N stretching (asymmetrical and symmetrical), and the band at 949 cm^{-1} corresponded to P-O-C stretching vibration in the side chain. The band at 774 cm^{-1} was assigned to P=N symmetrical stretching in the PBPP main chain [18-21]. The presence of P=N and P-O-C peaks confirms PBPP incorporation into the LDPE blend composites.

Band intensities at 1263 cm^{-1}, 1176 cm^{-1}, and 949 cm^{-1} increased with PBPP content due to higher functional group concentration.

B. Morphology Observation

Figure 2 shows fractured surface morphologies of MH-LDPE blend composites with different PBPP loadings. Electron micrographs reveal MH particles of one micrometer or less dispersed throughout the matrix. In Fig. 2(a), numerous gaps exist between MH particles and the LDPE matrix due to incompatibility, attributed to the polar nature of MH particle surfaces containing hydroxyl groups versus the non-polar LDPE matrix. Figures 2(b) and 2(c) show these gaps disappearing with PBPP addition. At low PBPP loading (Fig. 2b), the PBPP matrix is not clearly visible and appears surrounded by MH particles. The PBPP matrix becomes observable at higher loadings (Fig. 2c), suggesting incompatibility with the LDPE matrix. Figure 2(d) shows the cross-section morphology of sample PE-20-12.5 after ethanol washing. Cavities are observed because PBPP dissolves in ethanol while LDPE and MH do not, within short periods (e.g., 5 minutes). Gaps between MH particles and the LDPE matrix are also visible, proving that PBPP covers the MH particles. SEM analysis reveals that PBPP exists between MH particles and the LDPE matrix, potentially modifying interfacial interactions.

C. MFR Study

Melt flow rate (MFR) was used to characterize processing properties. Figure 3 [Figure 3: see original paper] shows MFR variation with PBPP content at constant 20 phr MH loading. MFR increased with increasing PBPP content while density remained constant. As shown in Fig. 2(c), with the PBPP matrix covering MH particles, frictional resistance and apparent viscosity in the flow cell decrease [22]. Figure 3 demonstrates that increasing PBPP content raises the melt flow rate, with significant increases above 5.0 phr PBPP due to the formation of continuous PBPP phases in the LDPE matrix.

Increased MFR values indicate lower energy consumption during blending extrusion and subsequent molding, thereby improving production efficiency. This result contrasts sharply with the addition of inorganic flame retardants alone, which typically causes severe processing performance deterioration. However, excessive PBPP addition leads to rapid deterioration of mechanical properties. After careful consideration, 5.0 phr PBPP content is deemed appropriate.

D. Mechanical Properties

Elongation at break increased as PBPP content rose from 0 to 5.0 phr (Fig. 4 Figure 4: see original paper). When PBPP was added to MH-LDPE blends, the PBPP matrix covered MH particles (as seen in SEM images), reducing frictional forces during stretching and increasing elongation at break. However, further PBPP content increases caused decreased elongation at break because the PBPP phase deteriorated the continuous LDPE phase. This trend agrees with previous reports [23]. Tensile strength (Fig. 4(b)) showed a downward trend with increasing PBPP content because the PBPP matrix covering MH particles reduced friction between MH particles and the LDPE matrix. At 200 kGy absorbed dose, blend composite elongation was lower than at 0 kGy, while tensile strength was higher due to LDPE crosslinking.

This study found that elongation at break varied significantly with PBPP content, reaching a maximum at approximately 5.0 phr for both 0 kGy and 200 kGy doses. Therefore, 5.0 phr PBPP is appropriate for LDPE blend composites containing 20 phr MH.

E. Flammability

Flammability was evaluated by LOI and UL-94 burning tests, with results listed in Table 2 . LOI values increased with MH loading level. PBPP-containing composites showed higher LOI than non-PBPP composites. UL-94 data indicate that only PE-50-12.5 achieved V2 rating without molten drops during combustion, demonstrating PBPP' s anti-dripping capability.

During combustion, MH undergoes decomposition and dehydration reactions. MH particles reduce burning material surface temperature by absorbing heat from the polymer surface. Steam produced from MH dehydration dilutes combustible gas and oxygen concentrations. After decomposition, dense MgO residues deposit on the polymer matrix surface, providing heat insulation, oxygen barrier effects, and smoke suppression. Additionally, MH can improve polymer surface char formation [24–29].

PBPP offers numerous advantages: halogen-free, low toxicity, low smoke generation, good UV stability, and excellent flame retardant efficiency. When subjected to heat or combustion, phosphorus-containing compounds convert to phosphorus oxygen acids through thermal decomposition. The generated metaphosphoric acid forms a stable polymeric phosphatic acid that prevents the composite material surface from exposure to oxygen and combustibles. Additionally, heated phosphorus compounds emit PO • radicals that capture OH • , H • , and other radicals in the combustion chain reaction [30–33]. Nitrogen in PBPP produces non-combustible gases (NH_3, N_2, NO, NO_2, etc.) through decomposition, diluting oxygen and combustible concentrations while absorbing heat. This process reduces substrate surface temperature. Meanwhile, nitrogen oxides from decomposition can capture free radicals and inhibit combustion chain reactions in polymer composites. Therefore, PBPP achieves flame retardancy through gas-phase cooling, insulation formation, and radical chain reaction termination [34–36].

F. Property Changes After Irradiation

The PBPP polymer chain contains a conjugated structure of repeating -P=N- units with numerous benzene rings in side chains. Benzene rings are conjugated structures with rich electron clouds that can resist and scatter electron beams. Material anti-irradiation properties were investigated through elongation at break measurements. Figure 5 Figure 5: see original paper shows elongation results for PE-50-0 and PE-50-12.5 samples at different absorbed doses. PBPP-containing blend composites exhibited significantly higher elongation than non-PBPP composites. The PE-50-12.5 blend composite reached maximum elongation at 200 kGy. Cross-linking dominated in the LDPE matrix at 200 kGy, after which degradation increased with absorbed dose. Tensile strength (Fig. 5(b)) increased with absorbed dose, though PBPP-containing composites showed lower tensile strength than non-PBPP composites, as explained by data in Fig. 4(b).

To illustrate PBPP' s role during irradiation, thermogravimetric analysis was employed. Figure 6 Figure 6: see original paper shows a two-step degradation process (385-420 °C and 430-505 °C). The first degradation step corresponds to PBPP and the second to LDPE. A significant change occurred in the first degradation step: PBPP content decreased as dose increased from 0 to 1000 kGy. Furthermore, the degradation temperature range shifted from 385-420 °C to 365-400 °C at 1000 kGy due to PBPP' s lower molecular weight after irradiation. DTG results demonstrate that PBPP protects the LDPE polymer matrix through self-consumption.

IV. Conclusion

The effects of PBPP in MH-LDPE blend composites on flame retardancy and electron beam irradiation resistance were examined. Melt flow rate (MFR) and scanning electron microscopy (SEM) characterized processing properties and morphology, while mechanical testing and TGA analyzed irradiated blend composites. The main conclusions are:

1. As PBPP content in the blend increases, melt flow rate increases, indicating that PBPP presence favors interfacial interaction between LDPE and MH.
2. Based on elongation at break values at different ratios, the appropriate PBPP content is approximately 5.0 phr at 20 phr MH loading.
3. Limiting oxygen index (LOI) values increase with PBPP content.
4. PBPP positively affects electron beam irradiation resistance through self-consumption, protecting the polymer matrix.

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