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Overview of Graphene Applications and Research in Flame Retardant Polymers

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Abstract:

The domestic and international research status of graphene flame retardant technology in polymer composites in recent years is reviewed, the research mechanism of graphene flame retardant technology in polymer composites and the preparation method are summarized, and the application and research overview of graphene in flame retardant polymers are summarized and prospected.

Keywords: Graphene; Novel composite flame retardant; Flame retardant mechanism and Modification

1.Introduction:

Polymer materials are widely used in aerospace, electrical and electronic, construction and transport fields due to their light weight, easy processing and multifunctionality, but their flammability and burning accompanied by molten droplets, smoke and toxic gas release and other problems have seriously restricted their safety and application range. Traditional flame retardants such as halogenated compounds have high flame retardant efficiency, but there are toxicity and environmental hazards, while non-halogenated flame retardants such as metal hydroxides, phosphorus and nitrogen flame retardants have become a hot spot of research due to their environmental friendliness [1]. However, single flame retardants often face the problems of high addition amount, poor compatibility, and insufficient smoke suppression effect, and there is an urgent need to develop efficient and environmentally friendly synergistic flame retardant systems. Graphene, as a typical two-dimensional nanomaterial, has a high specific surface area (theoretical specific surface area of 2630 $m^2 \cdot g^{-1}$), excellent thermal stability and unique lamellar barrier effect, which provides a new idea for polymer flame retardant modification. Its nanolayer structure can form a dense carbon layer during the combustion process, inhibiting heat and oxygen transfer, while adsorbing combustible volatiles and slowing down the combustion process. However, the compatibility of graphene with polymer matrix is poor, and the flame retardant effect is limited when it is used alone, so it needs to be modified by functionalization or compounded with other flame retardants to build a synergistic system in order to bring out its potential advantages.

In recent years, significant progress has been made in the synergistic flame retardant research of graphene and its derivatives (e.g., graphene oxide GO, reduced graphene oxide rGO)[2] with phosphorus and nitrogen flame retardants, metal hydroxides, and layered bimetallic hydroxide (LDH). For example, Chen Qi [4] et al. pointed out that the two-dimensional layered structure of graphene can promote polymer dehydration into carbon, and synergistically with phosphorus and nitrogen systems to form a 'physical barrier + catalytic carbon formation' dual role in epoxy resins to increase the amount of residual carbon by 25% to 30%, and the thermal stability is significantly improved. In terms of preparation methods, in situ polymerisation, solution blending and melt blending are widely used to construct graphene-based flame retardant composites. Hu Hongliang[5] et al. summarized the graphene and metal hydroxide synergistic

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system, and found that the lamellar structure of graphene can enhance the dispersion of magnesium hydroxide (MH) and aluminium hydroxide (ATH) to form a 'nano-restricted domain effect', which synergistically improves the carbon formation rate and smoke suppression performance of the composites. In addition, the composite of graphene and phosphorus-nitrogen flame retardants not only strengthens the flame retardant effect through gas-phase dilution (release of flame-retardant gases such as N_2 and CO_2) and condensed-phase carbon layer, but also endows the material with additional functions such as antistatic, e.g., the surface resistance of the CN-rGO-modified PLA is reduced to 10^8 Ω, which meets the requirement of antistatic materials.

Despite the excellent potential of graphene-based flame retardant systems, their large-scale applications still face challenges, such as controllable functionalization modification of graphene, low-cost macro-preparation, optimization of compatibility with different polymer matrices, and analysis of synergistic mechanisms under complex combustion environments. In this paper, we synthesize the research results in recent years, and review the construction of graphene-based synergistic flame retardant systems, flame retardant mechanisms, and multifunctional applications, with a view to providing references for the development of high-performance, environmentally friendly polymer flame retardant materials.

2. Study of graphene-mediated synergistic flame retardant

Flame retardants can be divided into organic flame retardants and inorganic flame retardants according to their chemical composition, and into reactive and additive types according to their method of use. Representative flame retardants include bromine, phosphorus-nitrogen and phosphorus flame retardants, as well as aluminum hydroxide, magnesium hydroxide and antimony trioxide [6]. The composite system formed by graphene and other flame retardants can not only significantly enhance the flame retardant properties of the materials, but also reduce the amount of certain components in the synergistic system, thus optimizing the cost control, environmental requirements and safety standards. Currently, researchers have carried out extensive studies on graphene synergistic systems, focusing on the synergistic flame retardant mechanisms between graphene and its derivatives, metal hydroxides, phosphorus and nitrogen flame retardants, and various types of graphene-based/polymer flame retardant materials.

2.1 Flame retardant mechanism of graphene/metal hydroxide

Metal hydroxides (such as aluminium hydroxide (ATH), magnesium hydroxide (MH), layered double hydroxide (LDH), etc.) typically exhibit excellent thermal stability, low smoke generation, and non-toxic properties. Compared to halogen-based and phosphorus-based flame retardants, metal hydroxides are more environmentally friendly, as they do not produce toxic by-products or secondary pollutants during the flame retardation process. As a result, they have garnered significant attention in recent years. However, when used alone, they have drawbacks such as low flame-retardant efficiency and the need for higher addition levels, and they can significantly degrade the mechanical and processing properties of the flame-retardant polymer matrix. Therefore, they are typically modified or blended with other flame retardants to enhance their flame-retardant efficacy [7].

According to Yang Feihao's research [8], two types of graphene-based flame retardants were synthesised using nanohybrid technology and applied to epoxy resin (EP) transition metal hybrid flame retardants (EGOPC): copper compounds were grafted onto the surface of graphene oxide (GO) using ethylenediamine and phenyl phosphoric acid dihydrogen, forming EGOPC. Synergistic mechanism: Copper catalyses CO oxidation and promotes char formation, while phosphorus-nitrogen compounds exert gas-phase flame retardant effects. Optimal ratio: At 3 wt% EGOPC, the limiting oxygen index (LOI) of the EP composite material reached 29.1%, the peak heat release rate (PHRR) decreased to 925.0 kW/m² (a 36.5% reduction compared to pure EP), CO generation rate decreased by 35.4%, and crosslinking density increased by 42.1%. Mesoporous SiO_2 hybrid flame retardant (SPGP): Phospholipid compounds are used to coat mesoporous SiO_2 and polyphenylene diamine grafted onto GO surfaces, forming SPGP. Synergistic mechanism: The physical barrier effect of mesoporous SiO_2 and the condensed-phase flame retardant effect of phosphorus/silicon elements synergistically enhance the density of the char layer. Optimal ratio: At 3 wt% SPGP, the total heat release rate (THR) and total smoke volume (TSP) were reduced by 48.8% and 20.97%, respectively, compared to pure EP; at 1 wt% SPGP, the impact strength reached 45 kJ/m² (a 100% increase), and the glass transition temperature (Tg) rose to 166.7°C. Both types of flame retardants significantly enhance the flame retardancy, smoke suppression, and mechanical properties of EP through multi-element synergistic effects (phosphorus/nitrogen/copper or phosphorus/silicon). EGOPC focuses on gas-phase-solid-phase synergistic flame retardancy, while SPGP reinforces the

physical barrier effect of the char layer. Li Kun [9] utilised the tunable interlayer structure of layered double hydroxides (LDH) to prepare flame-retardant epoxy resin (EP) composites via two nano-structural design strategies: Interlayer composite design: Based on the 'memory effect' of LDH, phosphomolybdate anions ($PMoO_4^{03-}$) were substituted for interlayer anions to produce LDH-MoP. Interlayer composite design: Based on charge characteristics, positively charged LDH interlayers were electrostatically composite with negatively charged zirconium phosphate (α-ZrP) layers to produce LDH/α-ZrP(U). Both modified LDHs were composite with EP at a 3% mass ratio to achieve synergistic flame-retardant effects: LDH-MoP/EP: limiting oxygen index (LOI) reached 32.2% (pure EP was 23.8%), UL-94 V-0 rating (no dripping); Peak heat release rate (PHRR) decreased by 34.9%, and total smoke release (TSR) decreased by 33.8%; Catalysed the formation of a dense, highly graphitised carbon layer, with char residue increased by 8.5%. LDH/α-ZrP(U)/EP: LOI increased to 31.7% (pure EP is 23.7%), UL-94 V-1 rating (no dripping); PHRR decreased by 39.1%, peak carbon monoxide release rate (PCOPR) decreased by 42.1%; residual carbon content increased by 11.6%, with a complete and dense carbon layer. Through the interlayer/plate nanostructure design of LDH, synergistic flame-retardant effects between LDH and $PMoO_{40}^{3-}$ or α-ZrP were achieved at a low addition level of 3%, significantly enhancing the flame-retardant and smoke-suppressing performance of EP, demonstrating its high-efficiency flame-retardant potential. Dong[10] et al. prepared Zn-Al layered double hydroxide anchored reduced graphene oxide (Zn-Al/LDH@RGO) hybrid materials to enhance the fire-resistant performance of water-based epoxy composite coatings. The layered Zn-Al/LDH and RGO exhibit a synergistic effect in the epoxy resin matrix: Zn-Al/LDH releases non-flammable gases such as CO_2 and water vapour upon combustion, diluting the concentration of flammable substances and cooling the matrix; RGO provides a high-barrier physical barrier, enhancing the strength of the char layer and inhibiting the escape of pyrolysis products. Experimental results indicate that the optimal ratio is 1.5 wt% Zn-Al/LDH@RGO; fire-resistant performance is improved: the lowest back temperature (154.3°C) (compared to 264.8°C for pure EP); the highest expansion rate (12.13), with the char layer expanding to a height of 16.5 mm; highest char residue rate (32.2%), and significantly enhanced thermal stability (T_{max} = 389.6°C). This synergistic system significantly improves thermal insulation and oxygen barrier efficiency by forming a dense honeycomb-like carbon layer and a metal oxide/phosphate-enhanced barrier, demonstrating its highly efficient fire-resistant performance.

2.2 Flame retardant mechanism of graphene/phosphorus-nitrogen flame retardant

The composite system of graphene and phosphorus-nitrogen flame retardants (expanding flame retardants, IFR) demonstrates significant synergistic effects in the field of polymer flame retardancy. This system achieves efficient flame retardancy through a dual mechanism: on the one hand, phosphorus-nitrogen flame retardants decompose under heat to produce acidic substances that catalyse the dehydration of polymers into char, while simultaneously releasing inert gases (such as NH_3) to expand and form a porous char layer, thereby isolating heat and oxygen; on the other hand, graphene, with its high specific surface area and layered structure, constructs a physical barrier in the condensed phase, inhibiting the diffusion of flammable gases and enhancing the density of the carbon layer, thereby creating a 'maze effect.' Experiments show that this synergistic system significantly improves flame retardant performance—for example, adding 2 wt% graphene and IFR to polypropylene (PP) reduces the peak heat release rate (PHRR) and total heat release (THR) by 74.4% and 14.8%, respectively (Yuan et al.); and introducing phosphorus-nitrogen functionalised graphene (4 wt%) into epoxy resin (EP) reduces THR by 30.2% (Yu et al.). Additionally, graphene promotes the graphitisation of char residue, increasing the char residue rate (e.g., 22.19% in the EP system), while the porous carbon layer of IFR and the barrier effect of graphene jointly suppress smoke release, conferring the system with environmentally friendly properties such as low smoke, halogen-free, and low toxicity.

Trubachev[11] et al. developed a composite material system with both high flame retardancy and mechanical properties by blending bio-based phosphorus-containing benzimidazole monomers (CBz) with graphene into glass fibre-reinforced epoxy resin (GFRER). Among these, CBz (20 wt%) catalyses the conversion of high-molecular-weight epoxy decomposition products (e.g., bisphenol-A) into phenolic compounds in the condensed phase, promoting char formation and accelerating the early release of low-molecular-weight products (e.g., CH_3^+) into the gas phase, thereby reducing the supply of fuel for subsequent combustion. This increases the limiting oxygen index (LOI) to 28.5% (a 5.4% improvement over the baseline sample) and achieves UL-94 HB self-extinguishing performance; Graphene (2 wt%) leverages its high specific surface area to adsorb large-molecule pyrolysis products, prolonging their decomposition time in the condensed phase and enhancing the physical barrier effect of the carbon layer. When combined (2 wt% graphene + 18 wt% CBz), they exhibit significant synergistic effects: the peak tensile load reaches 15.5 kN (a 32.5% increase),

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the fracture displacement increases by 122%, while maintaining the LOI at 26.1%. This study revealed through in situ mass spectrometry thermal analysis (DMSTA) that CBz and graphene jointly reduce the flammability of the material by regulating the pyrolysis pathways and product release kinetics, respectively, providing a new strategy for the design of high-performance flame-retardant composites. Ding Fang [12] et al. achieved efficient flame retardancy and smoke suppression in polyester fabrics by constructing a composite flame-retardant system based on phosphorus and nitrogen elements. The study employed layer-by-layer (LbL) self-assembly technology to alternately deposit gelatin/montmorillonite (G/MMT) polyelectrolyte-nano-clay composites and gelatin/phytic acid (G/PA) polyelectrolyte composites onto the surface of alkali-treated polyester fabrics, forming a phosphorus-nitrogen/clay flame-retardant system. This system leverages the synergistic flame-retardant mechanism of montmorillonite's layered barrier effect and phytic acid's catalytic carbonisation action, significantly enhancing the fabric's thermal stability and smoke suppression capabilities. Experiments showed that when the gelatin concentration in the G/MMT suspension was 2.0 wt% (sample PET-G/MMT@G/PA-2), the polyester fabric's limiting oxygen index (LOI) reached 32.4%, with no melting droplets during vertical burning and instantaneous self-extinguishing after the flame was removed during horizontal burning testing. Cone calorimetry tests showed that the peak heat release rate (pHRR) and total smoke release (TSP) were reduced by 43.7% and 22.5%, respectively, compared to the original fabric; the sample with a gelatin concentration of 1.5 wt% (PET-G/MMT@G/PA-1.5) exhibited superior smoke suppression, with a TSP reduction of 63.1%. Additionally, the flame-retardant system maintained a LOI of 23.5% after five water washes, demonstrating its excellent durability. Ma Wenjing[3] et al. synthesised a graphene-loaded phosphorus-nitrogen composite flame retardant (CN-rGO) and applied it to the flame-retardant modification of polylactic acid (PLA). The flame retardant was prepared using 2-carboxyethylphenyl phosphonium chloride (CEPPA), carbonamide, and reduced graphene oxide (rGO) as raw materials, and PLA/CN-rGO composite materials were obtained via melt blending. Experimental results showed that CN-rGO exhibits high-efficiency flame retardancy: when only 1% was added, the limiting oxygen index (LOI) of PLA increased from 20% to 37%, reaching the flame-retardant grade; the vertical burning grade was V-0 (self-extinguishing upon removal from flame), with no melting droplets; cone calorimetry tests showed that the peak heat release rate (PHRR) and total heat release (THR) decreased by 40.2% and 23.1%, respectively. Mechanistic analysis indicates that CN-rGO decomposes in the gas phase to produce flame-retardant gases that dilute oxygen, and in the condensed phase, it synergistically forms a dense carbon layer with rGO to block heat and oxygen transfer, thereby exerting a dual flame-retardant effect. Additionally, CN-rGO confers anti-static properties to PLA, reducing the surface resistance of the composite material to 10^9 Ω, achieving anti-static grade.

3. Preparation method of graphene synergistic flame retardant polymer composite materials

3.1 In-situ Polymerisation Method

The in-situ polymerisation method is a preparation technique that involves blending monomers or prepolymers with graphene and its derivatives, and introducing an appropriate amount of initiator to trigger monomer polymerisation. During this polymerisation process, the resulting polymer can intercalate between graphene layers, thereby achieving uniform dispersion of graphene within the composite material system and producing graphene nanocomposites. Mao Henan [13] et al. dispersed functionalised graphene oxide (GO) or graphene in polymer monomers, achieving uniform dispersion of nanosheets and forming strong interfacial chemical bonds during polymerisation, significantly enhancing the overall performance of the composite materials. For example, in the GO/polyimide system, when the GO mass concentration reaches 10%, the modulus and fracture strain of the composite film are doubled compared to pure polyimide; the conductivity of graphene/polystyrene composites prepared via in situ emulsion polymerisation is significantly enhanced; while in the GO/polyurethane system, the chemical bonding between isocyanate and GO hydroxyl groups effectively improves the material's thermal stability and mechanical properties. Additionally, this method can be applied to systems such as epoxy resin, polyaniline, and polyvinyl alcohol, demonstrating universal advantages in enhancing polymer conductivity, mechanical strength, and thermal stability. The core mechanism lies in the enhanced stress transfer efficiency between nanofillers and the polymer matrix due to the in situ formation of interfacial chemical bonds. Ko Ko[14] et al. used an in situ polymerisation method, with reduced graphene oxide (rGO) as the substrate, to prepare spherical (0-D), tubular (1-D), and sheet-like (2-D) polyaniline (PANI) using an in situ polymerisation method, with reduced graphene oxide (rGO) as the substrate. They then obtained rGO-PANI nanocomposites and studied their interfacial interactions and electrical

properties. The study found that the interface interactions between 0-D spherical PANI (PANI_H_2SO_4) and rGO were superior to those of 1-D tubular PANI (PANI_CH_3COOH) and 2-D sheet-like PANI (PANI_DW), and these differences in interface interactions were closely related to the electrical conductivity of the composite materials. Experimental results showed that the electrical conductivity of rGO-PANI_H_2SO_4 was 3.34×10^{-4} S, approximately 1,000 times that of rGO-PANI_CH_3COOH (6.52×10^{-7} S) and approximately 10^7 times that of rGO-PANI_DW (6.89×10^{-11} S), demonstrating its superior electrical performance.

3.2 Emulsion Polymerisation Method

In situ emulsion polymerisation is a method for preparing polymer/graphene composite materials. This method first mixes polymer monomers (or prepolymers), emulsifiers, graphene derivatives, etc. During this process, the monomers adsorb onto the surface of the graphene derivatives through non-covalent interactions such as hydrogen bonds or π-π interactions. Subsequently, the monomers are initiated to undergo free radical polymerisation reactions, ultimately forming the composite material [15]. Kang Wei[16] et al. modified reduced graphene oxide (RGO) using diethylaminoethyl methacrylate (DEAM) and dimethylaminoethyl methacrylate (DMAEMA) as modifiers, and then prepared poly(methyl methacrylate) (PMMA)/modified RGO nanocomposites via the emulsion polymerisation method. The amine groups of DEAM (DMAEMA) exhibit strong interactions with the surface functional groups of RGO, enabling complete encapsulation of RGO by PMMA, thereby significantly enhancing the performance of the composite material. Experimental results indicate that modified RGO significantly improves the thermal stability, electrical properties, and mechanical properties of PMMA/RGO nanocomposites, with DEAM modification yielding the best results. The glass transition temperature (Ts) of PMMA/DEAM-RGO increased by 12.4°C compared to PMMA, the initial thermal decomposition temperature increased by 54.1°C, the electrical conductivity reached 3.272×10^{-3} S/m, the impact strength increased by 25.82% compared to pure PMMA, and the tensile strength increased by 120.35%, demonstrating its excellent comprehensive performance. Alizade[17] et al. used the emulsion polymerisation method, with hydrogen phosphate ions as templates, acrylic acid and divinylbenzene as functional monomers and crosslinking agents, respectively, to prepare hydrogen phosphate-ion-imprinted polymers in a water-in-oil emulsion stabilised by hexadecyl trimethylammonium bromide. These were then mixed with graphite, graphene, and n-octadecane to prepare graphite paste potentiometric electrodes with selective recognition capabilities. The addition of graphene improved the electrode's charge conductivity, synergising with the imprinted polymer to significantly enhance the electrode's response performance towards hydrogen phosphate ions. Experimental results indicated that the optimal electrode composition was: 25.0% graphite, 31.2% imprinted polymer, 6.2% graphene, and 37.5% binder. The electrode prepared with the optimal composition exhibits a linear response range of 1×10^{-5} to 1×10^{-1} mol/L, a Nernst slope of 29.8±0.4 mV·decade^{-1}, a response time of 10 s, and a detection limit of 5.0×10^{-6} mol·L^{-1}, demonstrating its excellent selectivity and sensitivity towards hydrogen phosphate ions.

3.3 Melt Blending

Melt blending is a commonly used method for preparing polymer/graphene composite materials. This method typically involves mixing the polymer matrix, graphene (or its derivatives), and necessary additives (such as compatibilisers) in a molten state using equipment such as a screw extruder. During this process, mechanical shear forces gradually exfoliate and disperse graphene layers within the polymer matrix, while polymer chains may form interfacial bonds with graphene surface functional groups via van der Waals forces, hydrogen bonds, or chemical bonding. Jiang et al. [18] prepared graphene oxide-silica (GO-SiO_2) nanohybrids by grafting silica (SiO_2) onto the surface of graphene oxide (GO) via tetraethoxysilane (TEOS) hydrolysis, and then used the melt blending method to prepare graphene oxide-silica/polylactic acid (PLA) composites with different grafting rates. SiO_2 forms covalent bonds with the oxygen-containing functional groups on the GO surface, increasing the interlayer spacing of GO and effectively improving its dispersion in PLA. The two components exhibit synergistic effects to enhance the performance of the composite material. Experimental results indicate that the optimal grafting rate is 45.65%. At this grafting rate, the GO is well dispersed in the PLA in a single-layer form, with its crystallinity increased by over 2.17 times compared to pure PLA, significantly improved tensile strength, and an oxygen permeability coefficient reduced to 8.89×10^{-15} cm^3·cm·cm^{-2}·s^{-1}·Pa^{-1}, demonstrating significant improvements in crystallinity, mechanical properties, and barrier performance. Attar[19] et al. used a melt blending method to prepare nylon 6 nanocomposites by adding 20 wt% ethylene-octene copolymer (EOC), 3 wt% maleic acid-grafted EOC (EOC-g-MA), and different amounts of graphene nanosheets (GnPs) to nylon 6. EOC acts as a toughening agent to enhance material toughness, while GnPs increase stiffness. The synergistic interaction

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of these two components in the nylon 6 matrix enables balanced regulation of stiffness and toughness in the composite material. Experimental results indicate that the optimal ratio is 20 wt% EOC, 3 wt% EOC-g-MA, and 15 wt% GnPs. The composite material prepared at this ratio exhibits a fracture toughness of 19.8 kJ/m², surpassing the 13.9 kJ/m² of pure nylon 6, with a tensile modulus increased to 2.2 GPa, demonstrating its excellent comprehensive mechanical properties.

4. Summary and Outlook

Graphene, as an efficient flame-retardant synergist, demonstrates significant application potential in the field of polymer flame retardancy. Its flame-retardant mechanism primarily stems from its unique two-dimensional layered structure, which forms a dense physical barrier of carbon layers on the polymer surface during combustion. This carbon layer effectively blocks heat transfer and oxygen diffusion while adsorbing flammable volatiles, thereby delaying the combustion process. Additionally, graphene exhibits significant synergistic effects when blended with various flame retardants: Synergistic effects with metal hydroxides (such as magnesium hydroxide, Zn-Al layered double hydroxides): Synergistic effects with phosphorus-nitrogen-based flame retardants: The phosphorus component catalyses polymer carbonisation, the nitrogen component releases inert gases, while graphene reinforces the physical barrier function of the carbon layer, collectively significantly improving char residue rate and enhancing smoke suppression effects. Experimental studies have confirmed the excellent performance of graphene-based composite flame-retardant systems in various polymer matrices. For example, the epoxy resin composite material prepared by Guan[20]et al. (containing 68 wt% Al_2O_3, 7 wt% modified graphene nanosheets, and 5 wt% magnesium hydroxide) exhibited a high oxygen index (39%) and passed the UL-94 V-0 rating (no dripping). Adding just 1.5 wt% Zn-Al/LDH@RGO hybrid material to epoxy resin significantly enhances fire resistance, as evidenced by a significant reduction in back temperature to 154.3°C, an increase in expansion rate to 12.13, and an improvement in char yield to 32.2%. However, graphene still faces several challenges in flame-retardant applications: Dispersibility and compatibility issues: Graphene has poor compatibility with polymer matrices and tends to agglomerate, often requiring functionalisation modifications (such as grafting phosphorus-nitrogen groups or metal oxide coating) to improve its dispersibility. Need for synergistic efficiency optimisation: The flame-retardant efficiency of graphene alone is limited, and systematic optimisation of its ratio with other flame-retardant components is required to maximise synergistic effects. Insufficient understanding of mechanisms and safety assessment: A deeper understanding of flame-retardant mechanisms (such as the kinetic processes of carbon layer formation and the specific pathways of multi-component synergistic effects) is still needed; simultaneously, the potential toxicity and environmental compatibility of certain graphene-based composite flame-retardant systems require comprehensive assessment.

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