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Performance and preparation of modified pearlite thermal-insulating composites

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Abstract: In this article, three composites were prepared by filling the composition of hydrophobically modified pearlite with polyvinyl alcohol (PVA), urea-formaldehyde resin (UF), and melamine urea formaldehyde resin (MUF). Properties including combustion time, smoke density, oxygen index, and thermal conductivity, as well as the effects of component ratio on the combustion performance were determined for each of the three composites. The results indicated improved flame retardancy of all composite samples as a result of synergistic effects of the hydrophobically modified pearlite and the three binding agents. The PVA-based composite showed advantages in density, thermal decomposition, and smoke density, whereas UF-based and MUF-based ones showed good flame retardancy. Specially, the oxygen index of MUF-based composite material reached 52.5% (Level B1 flame retardancy).

Keywords: flame retardancy; melamine urea formaldehyde resin; pearlite; polyvinyl alcohol; urea-formaldehyde resin.

1 Introduction

Recently, there have been frequent reports of building fire accidents that are related to the external thermal insulating materials, often those organic in nature. It is, therefore, of great significance to improve the flame retardancy of current thermal insulating materials. Further, energy efficiency in buildings has attracted attention, with worldwide energy issues and the implementation of the sustainable development strategy of China (1–6). As a Level A insulating material (with good flame retardancy), pearlite is light, thermally insulating, and odorless, making it an attractive subject for intensive studies with applications in construction, food industry, and transportation. However, one drawback of pearlite is its surface moisture absorptivity, which leads to increased density and reduced thermal insulation. In this article, three inorganic-organic thermal insulating composites were prepared by filling the composition of hydrophobically modified pearlite with polyvinyl alcohol (PVA), urea-formaldehyde resin (UF), and melamine urea formaldehyde resin (MUF). The combustion performance, smoke density, oxygen index, thermal gravity, and thermal conductivity, as well as the effects of component ratio on combustion performance of the samples were investigated. The specific thermal conductivities and combustion performances of the samples were measured using standard experimental methods, allowing the discovery of a good balance between flame retardancy and thermal insulation. Compared to conventional thermal insulating materials that require a mixture of flame retardants, pearlite-based composites provide both excellent thermal insulation and reasonable flame retardancy.

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2 Materials and methods

2.1 Instrument

An electrical agitator (WH7401290, rotation rate = 200–3000 rpm) was from Weihua Instrument Pte. Ltd., Tianjin, China; an electronic balance (SE602F, accuracy = 0.1 mg) was from Jiazhan Instrument Pte. Ltd., Shanghai, China; 230 mm × 114 mm × 65 mm molds were customized; an oxygen index analyzer (HC-2CZ) was from Ascent Technologies Pte. Ltd., Nanjing, China; a building material combustion testing furnace (JCK-2) and a horizontal/vertical combustion analyzer (PMSC-3) were from ShineRray Instrument Pte. Ltd., Nanjing, China; a building material smoke density analyzer (JCY-3) was from Jiezhun Instrument Pte. Ltd., Shanghai, China; a thermogravimetry (TG) analyzer (TGA851e) was from Mettler-Toledo, Switzerland; and a thermal conductivity analyzer (HFM 436 Lambda) was from Netzsch Pte. Ltd., Shanghai, China.

2.2 Materials

PVA (polymerization = 1799) was from Chenqi Chemical Pte. Ltd., Shanghai, China; UF and MUF (analytical grade reagents) were from Sinopharm Chemical Reagent Co. Ltd. Pearlite (200 mesh) was from Incomperlite Pte. Ltd., Langfang, China; H_2O_2 (analytical grade reagent) was from Huadong Reagent Co. Ltd., Shenyang, China; silicone hydrophobic agent, of which the major functional ingredient was polyethylene oxide silane compounds, was from Defu Chemical Pte. Ltd., Wuhan, China.

2.3 Methods

2.3.1 Surface modification of pearlite

After rinsing using tap and distilled water, 100 g 200-mesh pearlite was dispersed in 7% H_2O_2 and stirred at 65°C for 4 h. The sample was then filtered and cooled to room temperature. Then the treated pearlite was mixed with silicone hydrophobic agent, stirred for 1 h, and dried in a thermostatic chamber (100°C).

2.3.2 Composite preparation

The surface-modified pearlite was mixed with either PVA, UF, or MUF and thoroughly stirred. Then the mixture was injected into the mold and allowed to cure forming each of the three composite samples in this way.

2.3.3 Evaluation of combustion performance of composites

The combustion performances of composites were evaluated based on corresponding national standards (7–11): GB/T 5480-2008 *Experimental methods for mineral wool and its products* for hydrophobic ratio (or water adsorption), GB/T 5990-2006 *Experimental methods for thermal conductivity of shaped insulating refractory products* for thermal conductivity, GB/T 2406.2-2009 *Oxygen index method for combustion performance of plastics* for oxygen index, GBT 8626-2007 *Experimental methods for flammability of building materials* for flammability, GB_T8323.2-2008 *Standards for measurement of plastic smoke density* for smoke density, and GB/T 2408-2008 *Combustion performance evaluation for plastics: horizontal and vertical method* for combustion performance.

3 Results and discussion

3.1 Effects of surface modification of pearlite on its hydrophobic ratio

The water absorption ratio of the surface-modified pearlite solution obtained under ambient conditions was measured and compared with conventional pearlite (Table 1).

As shown in Table 1, surface modification affects the hydrophobic ratio of pearlite. The transient water absorption ratio of conventional expanding pearlite can be as high as 344%; comparatively, surface-modified pearlite exhibits a significantly improved hydrophobic ratio (water absorption ratio as low as 1%) (Table 1). Surface-modified pearlite has hydrophobicity far beyond national standards, even when particle breakage – induced by mixing pearlite with inorganic binding agents and physical compression when molding are considered, making it a good energy efficient building material candidate. The hydrophobicity can be attributed to the strong adsorption capacity of the silicon hydrophobic agent; in particular, the decomposition products can adsorb on the surface of the mixture as expanding pearlite is added. After sublimation of the silicon hydrophobic agent, the sol covers the expanded pearlite as a molecular film. The hydrophobicity of expanding pearlite increases significantly due to the hydrophobic fluorocarbon chains in the silicon hydrophobic agent.

3.2 Effects of flame retardant composite on thermal conductivity of pearlite

Samples with different compositions were prepared and labeled as shown in Table 2 with their densities and thermal conductivity coefficients.

The composition ratio has a significant effect on the thermal conductivity of PVA, UF, and MUF composites, and their thermal conductivities are related to the composite

Table 1: Water absorption ratios of surface-modified pearlite and conventional pearlite. Groups A, B, and C were the three repeated experiments.

	Group A	Group B	Group C	Average water absorption ratio (wt%)
Pearlite	345%	319%	367%	344%
Surface-modified pearlite	1.03%	0.98%	1.00%	1.00%

Table 2: Mass ratios, densities, and thermal conductivity coefficients of different composite samples.

Mass ratio (%)	Thermal conductivity (w/mk)	Density (kg/m³)	Surface-modified pearlite	PVA	UF	MUF
1.1	0.043	168.1	90	10	0	0
1.2	0.047	174.4	85	15	0	0
2.1	0.051	269.0	90	0	10	0
2.2	0.054	281.6	85	0	15	0
3.1	0.068	284.2	90	0	0	10
3.2	0.074	320.5	85	0	0	15

density. As the content of binding agent increased, the composite density increased, resulting in increased thermal conductivity and reduced thermal insulating capacity. Among all samples, MUF-based composites showed the highest densities and highest thermal conductivities with the maximum (0.074 w/mk) at a MUF content of 15%. PVA-based composites showed the lowest densities, and the thermal conductivity was minimized (0.043 w/mk) at a PVA content of 10%.

3.3 Effects of binding agents on the combustion performance of pearlite

3.3.1 Effects of binding agents on the oxygen index of composites

The oxygen index is defined as the lowest oxygen ratio (in volumetric percentages) of oxygen/nitrogen required for a smooth combustion of a specific sample under prescribed conditions. According to GB 8624-2012 *Classification of combustion performances of building materials and its products*, the critical oxygen indices for Levels B1 and B2 flame retardancy for roof thermal insulating materials were 32% and 26%, respectively. Table 3 summarizes the oxygen indices of PVA-, UF-, and MUF-based (each at a composition ratio of 10%) composites. As observed, the oxygen indices of all composite samples exceeded 32%, indicating that all three composites obtained are flame retarding materials, which can be attributed to the high pearlite content (over 60%) in these composite samples. This level of flame retardancy is advantageous for industrial applications of these composites.

Table 3: Oxygen indices of PVA-, UF-, or MUF-based (10%) composites.

	PVA-based composite	UF-based composite	MUF-based composite
Oxygen index	33.5%	43.5%	52.5%

3.3.2 Effects of binding agents on horizontal and vertical combustion performance of composites

During the combustion of solid materials, the flame propagates over the surface, and the propagation rate (under prescribed conditions) is defined as the rate at which the flame travels. This parameter is a key indicator of the combustion performance of materials (12–14). According to GB/T 2408-2008 *Combustion performance evaluation for plastics: horizontal and vertical method*, the horizontal combustion performances of materials can be classified as Level HB, HB40, and HB75. In cases that no stable flame is observed, or in cases in which the front-end of the flame does not move more than 100 mm upon the removal of the ignition source, the material is classified as Level HB. The specific composite samples tested here had no flame combustions observed upon the removal of the ignition source, so these composites are classified as Level HB materials. The horizontal combustion performances of different composite samples are summarized in Table 4.

According to GB/T 2408-2008, the vertical combustion performances of materials can be classified as Levels V-0, V-1, and V-2. Composites based on UF and MUF are categorized as Level V-0 materials, whereas the vertical combustion performance of the PVA-based composite is beyond the current classification system and can only be categorized by its horizontal combustion performance.

As shown in Table 4, the composite base has a significant effect on combustion performance. The modified pearlite has excellent flame retarding capacity, as do the composites. For composites consisting of 40% binding agent and 60% modified pearlite, the flame disappears upon removal of the ignition source and the carbonization length is negligible, suggesting that the composites can be classified as Level B1 material.

Table 4: Horizontal and vertical combustion performances of different composite samples.

	PVA-based composite	UF-based composite	MUF-based composite
Horizontal combustion performance	HB	HB	HB
Vertical combustion performance	X	V-0	V-0

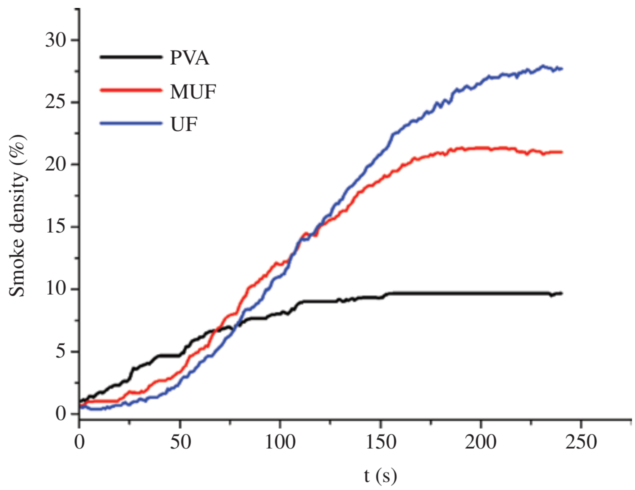

Figure 1: Smoke density of different composite materials.

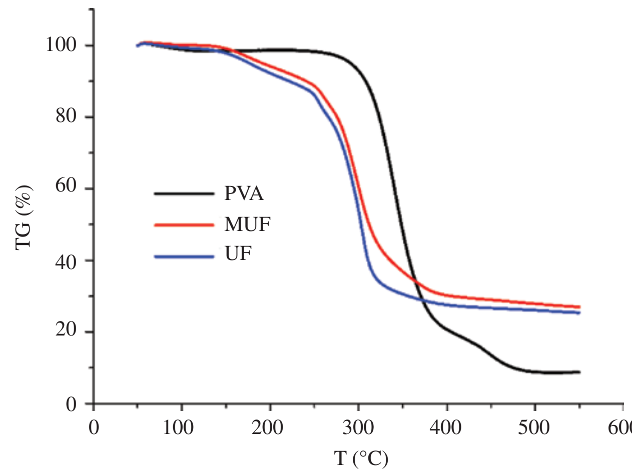

Figure 2: TG curves of different composite materials.

3.3.3 Effect of binding agents on smoke density of composites

As the toxic smoke released by combustions is the major cause of casualty in fire accidents, it is of great significance to measure the smoke density released by material combustion. The smoke densities of different composites (consisting of 60% modified pearlite and 40% binding agent) are summarized in Figure 1 to evaluate the effects of each binding agent on the combustion performance of pearlite-based composites. The smoke density of the PVA-based composite was the lowest, whereas that of the UF-based composite is the highest. The smoke density of UF may be attributed to the generation of soot, which is caused, more specifically, by the crosslinking of microresin molecules with unsaturated hydrocarbon molecules that are generated by UF matrix molecules during combustion, leading to the formation of macromolecules (soot). These macromolecules then interact with liquid phase molecules, and suspended solid particles and smoke are generated. Therefore, the resin content of composites has a significant effect on the smoke density.

3.3.4 Thermal decomposition of binding agents

The TG method refers to the measurement of mass (m) and temperature (T), where $m = f(T)$. Figure 2 shows the TG curves of different composite materials with temperature increasing at a rate of 20°C/min under dynamic nitrogen flow (50 ml/min). As observed, the TG curve of PVA is at the rightmost position, indicating high initiation and finishing temperatures of thermal decomposition. The TG curve of MUF is to the right of the UF curve, indicating that MUF has a reduced thermal decomposition rate and an extended thermal decomposition process.

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