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A hybrid intumescent fire retardant coating from cake- and eggshell-type IFRC

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ABSTRACT

In this study the applicability of flame retardant mixed with a carbon source (such as pentaerythritol) for the intumescent fire retardant coating (IFRC) daubed on the top of a piece of plywood was investigated. There are three kinds of flame retardants used in this study: (1) artificial mesophase graphite powder (MGP), (2) sericite ($Al_4(OH)_4(KAlSi_3O_{10})_2$), and (3) graphite. The desirable sizes of graphite, MGP and sericite were obtained by sieving. The graphite, MGP and sericite were characterized using a scanning electron microscope (SEM) and an energy dispersive spectrum (EDS). The IFRC, which consisted of 19.8% of flame retardant (or flame retardant mixed with carbon source), 15% of dehydrate agent, 18% of foaming agent, 7.2% of resin binder, and 40% of solvent, was prepared and daubed on the top of plywood. The fire protection capability of IFRC was tested using a flammability 45° tester. A conventional IFRC (with the carbon source) was also prepared to study the effect of adding the mixture of flame retardant and carbon source on the fire protection capability of IFRC. The microstructures of the conventional IFRC, the IFRC with flame retardant, and the hybrid IFRC (with flame retardant and carbon source) were inferred and demonstrated using SEM micrographs of the cross-section of three kinds of burnt IFRC. Most interestingly, the fire protection capability of the hybrid IFRC exceeds that of the conventional IFRC even though the percentage of carbon in the burnt hybrid IFRC is less than that in the burnt conventional IFRC.

1. Introduction

The flammability of wood limits its applicability because of the possible heavy casualties or the uncountable damage to the property caused by a fire accident. Therefore, improving the fireproof capability of wood (or wood-based material) has become an important issue that has attracted the attention of researchers. The surface treatment method called intumescent flame retardant coating (IFRC) is commonly used to protect a material against fire because it is a simple, efficient, and economical method of fire protection. In addition, it does not modify the intrinsic properties of the material [1–3].

The conventional IFRC, which consists of a carbon agent (such as pentaerythritol (PER)), a blowing agent (such as melamine (EN)), and an acid source (such as ammonium polyphosphate (APP)) [1–10], forms an intumescent char layer on the top of the material during heat exposure. However, the APP/PER/EN system normally produces a fluffier barrier of fire retardant (like a cake), which is easily penetrated by fire. Accordingly, flame-retardant fillers (such as expandable graphite [2,9], nano-sized magnesium aluminum-layered double hydroxides (nano-LDHs) [3], nano-sized silica (SiO_2) [4], 4A type zeolite [5], vitreous filler [6], and silicotungstic acid (SiW_{12}) [7]) were proposed to form a compact microstructure in the charred layer. Aside from this, Riva et al. [11] studied the thermal and combustion behavior of an intumescent fire retardant system based on Polyamide 6 (PA6) and Ammonium Polyphosphate (APP), which are used to improve flame retardant properties of poly(ethylene-co-vinyl acetate) (EVA) and are loaded with $Mg(OH)_2$ (MH).

The aforementioned flame-retardant fillers may increase the cost of IFRC. Therefore, expandable graphite was used as both the carbon agent and synergistic agent to promote the strength of the barrier of fire retardant and reduce the cost of IFRC [12,13]. In addition, Chou et al. investigated the feasibility of new flame retardants (such as artificial mesophase graphite powder (MGP), sericite ($Al_4(OH)_4(KAlSi_3O_{10})_2$), or MGP/sericite mixture) on IFRC. They observed that the mass ratio of MGP to sericite substantially affects the fire protection capability of IFRC, and the fire protection capability of IFRC with the flame retardant of the raw material is better than that of IFRC with the flame retardant of the sieved and surface-modified material [14]. However, this new system of fire retardant normally produces a thin shell barrier of fire retardant (like an eggshell), which does not have a porous structure to contain more nonflammable gases generated by the dehydrate agent and the foaming agent.

Consequently, the study of an IFRC obtained locally, which has a reliable capability of fire protection, is worthy of continued investigation. The purpose of this work is to study the feasibility of flame retardant (such as graphite, MGP, or sericite) mixed with carbon source (such as pentaerythritol) on the IFRC, and to propose a hybrid IFRC with a reinforced microstructure. The effect of the different kinds

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of flame retardant (or the mixture of flame retardant and carbon source) proposed herein on the fire protection capability of IRFC daubed on the top of the plywood was investigated using a flammability 45° tester.

2. Experimental apparatus and procedure

The experiments included: (1) preparing the flame retardants and measuring their characteristics; (2) preparing the IFRC and measuring its characteristics; and (3) conducting the fire protection tests of IFRC.

2.1. Preparing and measuring the flame retardants

The three kinds of flame retardants used in this study were: (1) artificial mesophase graphite powder (MGP) produced by China Steel Chemical Corp. with a true density of 2.224 g/cm^3, a particle size that ranges from 5 to 58 μm, and water content of 0.08%, (2) sericite ($Al_4(OH)_4(KAlSi_3O_{10})_2$) produced by Sunshine Mineral Co., Taiwan with a particle size that ranges from 1 to 53 μm and a melting point of 1260–1290 °C, and (3) graphite with a true density of 1.15 g/cm^3, a particle size that ranges from 5 to 100 μm, and water content of 0.12% (Table 1). A high-precision balance (EXCELL BH-150) with a measuring range of 0.005–150 g was used to weigh the powder of graphite, MGP, and sericite.

Fifty grams of MGP (graphite, or sericite) was sieved using an analytical sieve shaker (Retsch AS-200) with a sieve of 400 mesh and a duration of 20 min. The micrographs of the MGP, graphite, and sericite were obtained using a SEM (Jeol JSM-6380) with a magnification of 15–100,000×. An EDS (Horiba EX-200) was used to analyze the weight ratios of the elements in the MGP, graphite, and sericite. Moreover, a mixture of 85% of carbon source (such as pentaerythritol) and 15% of flame retardant (such as MGP, graphite, or sericite) was prepared.

2.2. Preparation and measurement of the IFRC

The IFRC with flame retardant (such as MGP, graphite, or sericite) or the mixture of flame retardant and carbon source was fabricated and daubed on a piece of plywood to test the fire protection. Table 2 presents the test conditions under which the IFRC was prepared.

The procedure for fabricating the IFRC with the flame retardant (or the mixture of the flame retardant and the carbon source) is as follows. (1) A total of 7.5 g of dehydrate agent (ammonium polyphosphate, $[NH_4PO_3]_n$) was mixed with 9 g of foaming agent (melamine, $C_3H_6N_6$), 5 g of pure water, and 10 g of alcohol and was stirred well until the formation of colloid; (2) the colloid prepared by step (1) was mixed with 9.9 g of the flame retardant (or the mixture of flame retardant and carbon source) using a mixer (Tokushu Kika) for 10 min with a rate of rotation of 1500 rpm; and (3) the glutinosity of 3.6 g of resin binder (water-based acrylic resin) was adjusted by the addition of 5 g of alcohol, and this diluted resin binder was then mixed with the colloid prepared by step (2).

The procedure of measuring the viscosity of IFRC is as follows: (1) 100 ml of IFRC was poured into a beaker; (2) the viscosity of IFRC was measured using a viscometer (Brookfield DV-E) with a rotation speed of 500 rpm. The viscosity of the IFRC used in this study was between 262.1 and 353.3 cP (Table 2). Moreover, a pH meter (Hanna Instrument pH-211) was used to obtain the pH of IFRC at room temperature of 25 °C. The pH of the IFRC used herein was between 7.8 and 8.6 at 25 °C (Table 2).

Table 1
Test conditions of preparing flame retardants.

	Graphite (%)	MGP (%)	Sericite (%)	Pentaerythritol (%)
A 1	100	–	–	–
A 2	–	100	–	–
A 3	–	–	100	–
B 1	15	–	–	85
B 2	–	15	–	85
B 3	–	–	15	85
C 1	–	–	–	100

Table 2
Test conditions of preparing the intumescent fire retardant coatings.

	Preparing IFRC					pH (at 25 °C)	Viscosity (cP at 25°C)
	20% Flame retardant/ carbon source	15% Dehydrate agent	18% Foaming agent	7% Resin binder	40% Solvent		
W1	–	–	–	–	–	–	–
W2	C 1	Ammonium polyphosphate	Melamine	Water-based acrylic resins	Alcohol/ Pure water (3:1)	7.8 ∫ 8.6	262.1
W3	A 1						335.3
W4	A 2						325.7
W5	A 3						353.3
W6	B 1						296.2
W7	B 2						285.7
W8	B 3						309.4

2.3. Fire protection test of the IFRC

Fifty grams of IFRC was daubed on the top of plywood with a size of 290 (L) × 190 (W) × 4 (H) mm using a home-made applicator (Fig. 1). The plywood with a layer of IFRC was then dehydrated in a furnace for 48 h with a temperature of 45 °C. After that, the thickness of IFRC on the top of the plywood was measured using a vernier. The thickness of IFRC was controlled and maintained at 0.25 mm in all 8 experiments.

A flammability 45° tester (Suga FL-45 M) was used to conduct the fire protection test of IFRC on the top of the plywood (Fig. 2). In each test, the duration, height, and maximum temperature of the flame were 120 s, 6.5 cm, and 600 °C, respectively. Aside from this, a Bunsen burner was used as the heat source (Fig. 2), and the maximum temperature of flame could be obtained within 10 s. Moreover, the durations of the remaining flame and the afterglow were determined. After the temperature of the plywood was lowered to room temperature, the mass burning percentage, the carbonized length, and the burned-through area of the plywood were measured. The image of the plywood with (or without) a layer of IFRC was then obtained using a digital camera (Panasonic DMC-LZ2). Furthermore, SEM (Jeol JSM-6380) was used to obtain the micrographs of the unburnt and burnt IFRC.

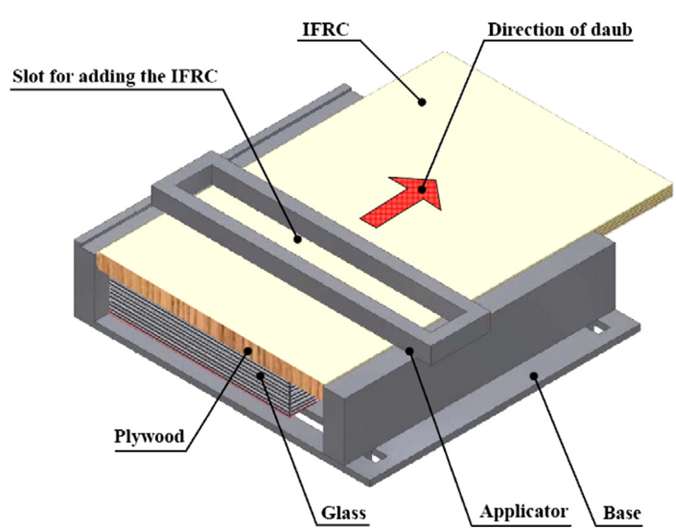

Fig. 1. Schematic of the apparatus of daubing IFRC on a plywood [14].