Development of advanced intumescent flame-retardant binder for fire rated timber door

Jessica Jong Kwang Yin¹, Yew Ming Chian ¹,*, Yew Ming Kun²

¹Department of Mechanical and Material Engineering, Lee Kong Chian Faculty of Engineering and Science, Universiti Tunku Abdul Rahman, Cheras 43000 Kajang, Malaysia
²Department of Civil Engineering, Lee Kong Chian Faculty of Engineering and Science, Universiti Tunku Abdul Rahman, Cheras 43000 Kajang, Malaysia

Abstract. Intumescent flame-retardant binder (IFRB) offers a great advancement for the most efficient utilization of a wide variety of passive fire safety system at the recent development. This article highlights the fire-resistance and thermal properties of the IFRB using Bunsen burner and thermogravimetric analysis. The five IFRB formulations were mixed with vermiculite and perlite for the fabrication of fire-resistant timber door prototypes. Additionally, the fire rated door prototypes were compared under 2 hours fire test. The prototype (P2), with a low density of 637 kg/m³ showed the superlative fire-resistance rating performance, resulting in temperature reduction by up to 58.9 °C, as compared with that of prototype (P1). Significantly, an innovative fire rated timber door prototype with the addition of formulating intumescent binder has verified to be effective in stopping fires and maintaining its integrity by surviving a fire resistance period of 2 hours.

1 Introduction

The passive fire protection (PFP) system plays an important role in fulfilling the fire safety building regulations to effectively stop the propagation of a fire. PFP system includes fire or smoke dampers, fire doors and firewalls. Therefore, with regards to this research, a new and innovative fire-resistant timber door is incorporated with the intumescent flame-retardant binder (IFRB) acts as an effective PFP system for building occupants to escape safely during the outbreak of fire by trapping the fire and smoke. The intumescent binder reacts under the influence of fire and swells to many times of its original thickness, and thus produces an insulation carbonaceous char or foam that protects the substrate from the effects of the fire [1].

In this study, intumescent binders and lightweight flame-retardant materials such as vermiculite and perlite are used to construct the lightweight fire-resistant timber door prototypes (density is about 600 ±50 kg/m³) for 2-hour fire rating. Intumescent paint is the recent trend in fire retarding product in construction building materials because of its many beneficial properties such as providing low-odor, lightweight, and environmentally friendly. Exhaustive investigations have revealed that the intumescent flame retardant coating has achieved good flammability, physical and chemical performances though many linger widely on steel structure applications [2-4]. Up to present, the performance of fire-resistant board has yet to be tested and investigated with the addition of intumescent binder for the development of fire-resistant timber door. Furthermore, this research project has also highlighted a huge potential of incorporating the intumescent materials, which consists of three main flame retardant additives, namely ammonium polyphosphate (APP) – acid source, pentaerythritol (PER) – carbon source and melamine (MEL) – blowing agents, into the fire door. These are mixed in a weight ratio of 2:1:1 and are to be bonded together with flame retardant fillers and vinyl acetate (VA) copolymer as well as vermiculite and perlite which react together to form a protective thermal barrier at high temperature in fire door [5]. It is important to note that flame-retardant additives are useful chemical compounds for fire retardant to provide varying degrees of flammability protection.

This project aims to design and fabricate the fire-resistant timber door prototypes using the formulating IFRBs to fulfill the 2-hours fire rating.

2 Materials and sample preparation

The first part of this project is to synthesize the water-based intumescent binder using a high speed disperse mixer for 30 minutes at room temperature until it is completely homogenous. The formulations of intumescent coatings were tabulated in Table 1. APP, PER, and MEL were used as flame retardant additives. The aluminium hydroxide, magnesium hydroxide, calcium silicate, chicken eggshell (CES) powder, calcium carbonate, and titanium dioxide were used as
flame-retardant fillers and vinyl acetate (VA) copolymer emulsion acts as water-based polymer binder. The fire resistance and thermal properties of the IFRBs were characterized and assessed through Bunsen burner test and thermogravimetric analysis.

Table 1. Compositions of the IFRB

| Samples Ingredients | Parts by weight for formulations (wt. %) |
|---------------------|----------------------------------------|
|                     | J1 | J2 | J3 | J4 | J5 |
| Flame-retardant additives |     |     |     |     |     |
| APP                 | 20 | 20 | 20 | 20 | 20 |
| PER                 | 10 | 10 | 10 | 10 | 10 |
| MEL                 | 10 | 10 | 10 | 10 | 10 |
| Vinyl acetate (VA) copolymer emulsion | 50 | 50 | 50 | 50 | 50 |
| Flame-retardant filler (pigment) |     |     |     |     |     |
| TiO₂                | 4.0 | 4.0 | 4.0 | 4.0 | 4.0 |
| Flame-retardant fillers |     |     |     |     |     |
| Al(OH)₃             | 3.0 | 3.0 | -  | -  | -  |
| Mg(OH)₂             | -   | -   | 3.0 | 3.0 | 3.0 |
| CaSiO₄              | 3.0 | -   | 3.0 | -   | -  |
| CES                 | -   | 3.0 | -   | 3.0 | -  |
| CaCO₃               | -   | -   | -   | 3.0 | -  |

The second part of this project is to design and fabricate the fire-resistant timber door prototype with a dimension of 300 mm (length) × 300 mm (width) × 50 mm (thickness) including 2 layers of plywood (thickness is 4 mm per layer). The fire protective performance of the fire timber door prototype was determined through a small-scale fire endurance test. Table 2 shows the details of the experimental prototypes.

Table 2. Specifications of prototypes

| Fire door prototype | Dimensions: 300 mm (L) × 300 mm (W) × 50 mm (T) | P1         | P2         | P3         | P4         | P5         |
|---------------------|------------------------------------------------|------------|------------|------------|------------|------------|
| Weight (g)          |                                                | 2279       | 2293       | 2300       | 2315       | 2318       |
| Density (kg/m³)     |                                                | 633        | 637        | 639        | 643        | 644        |

Moreover, the fire endurance test and the temperature rise test were conducted to compare and determine the best fire timber door prototype in terms of heat transmission and integrity failure or a significant leakage.

2.1. Bunsen burner test

The intumescent formulations were coated on a 100 mm × 100 mm × 3 mm steel plate with a thickness of 2 ±0.2 mm. The thermocouple plate is attached at the backside of the steel plate coated with intumescent flame-retardant coating and then connected to a digital handheld thermometer for temperature measurement.

Figure 1 shows the experimental setup of the Bunsen burner test for the coated samples. The sample was exposed to Bunsen burner flame spray gun blowtorch for one hour at a temperature flame of about 1000 °C. The Bunsen burner gas consumption is about 160 g/h and the distance between the sample and Bunsen burner nozzle is about 7 cm.

2.2 Thermogravimetric analysis

Thermogravimetric analysis (TGA) was carried out using a Perkin Elmer STA8000 model to determine the thermal degradation by calculating the residual weight of the intumescent coatings. About 5.8 mg of thin films were placed in a crucible and heated from 30 to 1000 °C at a heating rate of 20 °C/min under nitrogen gas.

2.3. Testing for fire resistant timber door prototype

2.3.1 Small scale fire test

The small-scale fire test was conducted on prototype 1–5, the intumescent binders for each prototype which is based on the formulation J1–J5 that provided in Table 1 as shown in section 2.1. After that, the fire protection performance of the prototypes was tested using the Bunsen burner at about 1000 °C for 2 hours. The temperature profile of the backside of the fire-resistant timber door prototype is measured and recorded using a digital handheld thermometer. Figure 2 shows a small-scale fire test experimental setup for the fire-resistant timber door prototype.
maintain its integrity when exposed to the 2-hour fire (at about 1000 °C) without an integrity failure or displaying a significant leakage [6]. Whereas, the temperature rise is also recorded over a 30-minute time interval until 120-minute, this is to examine the heat rise on the backside of the fire-resistant timber door prototype. The thermocouples are fixed at two points of the fire-resistant timber door prototype (centre (T1) and edge (T2)) to measure the transmission of heat from one point to another point of the prototype. This is to ensure that the prototype has taken adequate fire protection by reducing the heat transmission rate and flame propagation so that it can prolong the evacuation time for the building occupants if there is a fire.

3 Results and discussion

3.1 Bunsen burner test

The purpose of this specified test is to characterize the physical and chemical reactions of the char formation of the IFRB. The temperature profiles of the steel plates coated with five IFRB formulations (J1 – J5) and the bare steel plate are compared after the 60 minutes of fire testing. In this experimental work, the temperature of 400 °C is chosen as the critical temperature for the coated sample with IFRC [7]. The fire protection results of the coated steel plates are then plotted as a function of time and presented in Figure 3.

![Fig. 3. Evolution of temperature on the coated steel plates](image)

The first 10 minutes have showed that a similar pattern of the temperatures of the coated samples is below 300 °C. After that, the temperatures have started to fluctuate at 20 minutes due to the physical and chemical reactions of the coating formulations during the high temperature test. The temperature continued to rise for 30 minutes; it is observed that the coating J5 has started to rapidly increase until reaching 360 °C at 42 minutes. This may possibly be due to this coated sample has started to lose its adherence strength or interfacial bonding from the steel plate during the fire test. Whereas coating J1 has dropped rapidly after reaching 205 °C at 43 minutes, and this might due to the reducing of gas pressure from the Bunsen burner flame spray gun blow torch. It is also observed that the temperature of all the coatings has started to fluctuate again until reaching at 50 minutes. After 50 minutes of the test, the temperature has reached an equilibrium value and almost remained unchanged until the last stage of the test. The equilibrium temperature of coating J2 that is 217 °C is significantly lower than the other coatings and thus it has showed to be the best result in the fire protective performance. The char combined the positive effects of the flame retardant additives, intumescent paint and fillers (3 wt. % of Al(OH)3 and CES), promoting good fire protective barrier, and producing an excellent and the thickest char layer (i.e. about 6.0 mm) to block the fire while safeguarding the formation of a uniform foam structure which is in good agreement with other researchers, as highlighted by Wang and Yang, 2010 [8]. On the other observation, the coating J5 has the highest equilibrium temperature of 325 °C, reaching almost to the critical temperature (400 °C) after 40 minutes of the fire exposure. The equilibrium temperature of coating J5 might be due to its char layer showed porosity and lightness, resulting in the loss of adherence of the char to the steel plate or by a loss of cohesion of the char layer [9].

3.2 Thermogravimetric analysis

From the result obtained in Figure 4 under nitrogen gas, the thermal decomposition is mainly observed at the temperature in between 200-450 °C. However, the thermal decomposition can be occurred in different stages.

![Fig. 4. TGA curves of samples](image)

In this study, the general thermogram profiles are quite similar. However, the curves are found to be slightly shifted towards higher temperature ranges with increments in the heating rate. From 30-100 °C, around 2 % of the initial weight is loss. This might due to the possibly owing to the escape of moisture. Between 100-200 °C, J1 and J2 had slightly weight loss approximately 1 to 2 %, where J3, J4 and J5 had around 5 % of the initial weight is observed. Between 200-300 °C, a weight loss around 10 % of initial weight is observed. Between 300-450 °C, around 45 % of the initial weight loss is observed. Between 450-1000 °C, the weight loss around 10% of the initial weight is observed. This can be attributed from the degradative reactions occurring to the relatively stable components in the mixture. The residue weight of J1, J2, J3, J4 and J5 at 950 °C was 34.28 %,
34.55%, 32.77%, 33.82% and 31.34%, respectively. The TG curves demonstrated that the residue weight of coating sample J2 had the highest residue weight which indicated that the addition of aluminium hydroxide and CES fillers content in intumescent binder could enhance anti-oxidation of the coatings [9-11].

3.3 Testing for fire resistant door prototypes

This test is to characterize the fire protective performance of the fire-resistant timber door prototypes (P1-P5). The results of each sample plotted as a function of time are presented in Figure 5. The results show that the temperature of each fire-resistant timber door prototypes has increased gradually. From the results obtained, all the temperatures of prototypes increase significantly at the first 15 minutes revealing a similar pattern. This phenomenon is because of the good thermal insulation of vermiculite and perlite. These porous materials can dissipate the heat when exposed to fire. After 60 minutes, all prototypes were gradually increased until 120 minutes. This may be due to the physical and chemical reactions of the fire-resistant boards during the fire test. On top of that, P2 has the lowest temperatures of 140.7 °C and 32.0 °C at the T1 and T2, respectively. It has also indicated that this prototype resulted in the best fire protective performance in retarding the fire.

![Fig. 5. Evolution of temperature of fire-resistant timber door prototypes.](image)

4 Conclusions

From the series of experimental tests, the following conclusions can be derived based on the results presented in this paper:

1. The coating J2 with the combination of 3 wt. % aluminium hydroxide and 3 wt. % renewable CES bio filler have showed to be the best fire protective performance with an equilibrium temperature of 217 °C.

2. The TG curves demonstrated that the residue weight of coating sample J2 had the highest residue weight of 34.55 % at 950 °C, which indicated that the aluminium hydroxide and CES fillers content could enhance anti-oxidation of the coating.

3. The P2 prototype with the incorporation of J2 intumescent binder has also indicated the best fire protective result (temperature reduction by up to 58.9 °C), as compared to P5 prototype.

The selection of an appropriate combination of flame-retardant materials can directly affect the fire protective performance and thermal properties of the intumescent flame-retardant binder. As a result, the lightweight fire-resistant timber door prototype (P2) has indeed demonstrated to be more efficient in reducing the heat transmission by maintaining its integrity without showing a significant leakage against the 2-hour fire test.

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