

Article

Smoke Suppression Properties of Fe₂O₃ on Intumescent Fire-Retardant Coatings of Styrene–Acrylic Emulsion

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Abstract: The intumescent flame-retardant coatings were prepared using ammonium polyphosphate (APP), pentaerythritol (PER), melamine (MEL), styrene–acrylic emulsion, and iron oxide yellow (FeOOH) as the base material. A cone calorimeter (CCT), smoke density meter (SDA), and scanning electron microscope (SEM) were employed to investigate the smoke suppression and flame retardancy of FeOOH in intumescent fire-retardant coatings. The thermal degradation performance of intumescent fireproofing coatings with varying FeOOH content was investigated through thermogravimetric analysis (TGA). The structure of the carbon slag in the CCT test was analyzed using a scanning electron microscope (SEM). The results of the cone calorimeter (CCT) experiments demonstrated that FeOOH significantly reduced the heat release rate (HRR), total heat release rate (THR), smoke production rate (SPR), and total smoke release rate (TSR) of the coating, while simultaneously increasing the carbon residue rate of the coating. The smoke density analysis (SDA) results demonstrate that adding FeOOH can effectively reduce smoke generation, regardless of whether a pilot flame is used. TGA results demonstrate that FeOOH can enhance the weight of coke residue at elevated temperatures. SEM results indicate that incorporating FeOOH resulted in a more compact coke residue. According to these findings, among all the samples, those containing 2 wt% FeOOH showed low levels of HRR, THR, SPR, and TSR and high levels of SOD, which proves that FeOOH can be used as a smoke inhibitor in flame-retardant coatings.

Keywords: intumescent fire-retardant coatings; FeOOH; smoke suppression



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1. Introduction

It is a well-documented fact that a significant number of fire accidents caused by polymeric materials occur each year worldwide. These incidents result in a considerable loss of life and property, which has prompted concern among government regulatory bodies, consumers, and manufacturers alike [1]. It is now widely accepted that the primary cause of fires is not the heat itself, but rather the smoke and volatile compounds produced during combustion [2]. It is well documented that smoke, particulates, and certain toxic compounds (chiefly carbon monoxide) are responsible for over 70% of fire-related fatalities [3]. The dangers of smoke to human life are particularly evident in the initial phase of a fire, when light is obscured and gases and particulates scatter, leading to disorientation and panic [4]. This can result in irrational behavior, which in turn makes escape difficult and often fatal [5]. The two main aspects of the smoke hazard are the obscuration of exits and toxicity [6]. In addition, the thick smoke released by the polymer composite material greatly reduces visibility and prevents people from escaping the fire. In addition, specific issues such as corrosion, smoke emissions, and the toxicity of combustion products are of concern [7]. Therefore, it is necessary to investigate the adverse effects of fire smoke.

Fireproof coating is a simple and effective method to protect materials from fire and has been widely used [8]. The advantages of fireproof coatings are as follows: First, the

fireproof coating retains the inherent characteristics of the material, such as its mechanical properties. Secondly, applying for fireproof coatings is a quick and straightforward process. Third, they can be used on a variety of different substrates, including metal materials, polymers, textiles, and wood [1,9,10]. Intumescent fire-resistant coatings have gained considerable recognition worldwide for their use in protecting materials from fire, mainly due to their favorable environmental characteristics. The preparation of these coatings involves the use of carbonizing agents, acid sources, and blowing agents [11–14]. When exposed to fire, the coating expands and forms a protective layer that is many times thicker than the original coating. However, the burning process also produces a large amount of smoke, which can pose significant risks, including death. Therefore, it is necessary to effectively reduce the emissions of smoke and toxic gases. Many different compounds are used as smoke suppressants, such as alloys including antimony, zinc and copper (especially iron and molybdenum), organic and inorganic compounds, etc., which are widely used as flame retardants and smoke suppressants for organic polymers [15,16]. The mechanism of tin compounds used as halogenated polyester thermosetting flame retardants and smoke suppressors has been extensively studied [6]. Zinc borate (ZB) has been shown to be a flame retardant and smoke suppressor for PVC [17]. Subsequently, zinc hydroxystannate (ZHS) and zinc stannate (ZS) are highly effective flame retardants and are widely used in the fire protection of polymer materials [18]. In addition, many iron compounds can also significantly reduce the smoke produced by PVC combustion [19]. In addition, metal oxides, including molybdenum trioxide (MoO_3) and iron oxide (Fe_2O_3), have been shown to improve the thermal stability of the carbon produced by APP/PER/MEL flame-retardant coatings [1,20]. However, studies on the smoke suppression performance of FeOOH in intumescent fireproof coating systems are still lacking.

This paper presents a series of intumescent fire-retardant coatings prepared using PER as a carbonization agent, APP as the acid source, melamine as a blowing agent, and FeOOH as a smoke suppressant, respectively. In order to gain a deeper understanding of the smoke suppression effect of FeOOH in the intumescent fire-retardant coating, a series of studies were conducted using CCT, SDA, SEM, and TGA.

2. Materials and Methods

2.1. Materials

The styrene–acrylic emulsion (solid content 50.84%) was provided by the Qingdao Sea Chemical Research Institute. The ammonium polyphosphate (APP), pentaerythritol (PER), melamine (MEL), and iron oxide yellow (αFeOOH) (L1916, technical grade) were purchased from BASF Chemical Co. Ltd., Tianjin, China. The FeOOH is applied to automotive and industrial paints. The particle size of FeOOH is approximately 10 μm . The intumescent flame retardant (IFR) was obtained with a mass ratio of APP, PER, and MEL of 3:1:1. All other chemical reagents were without any purification.

2.2. Preparation of Intumescent Fire-Retardant Coatings

The intumescent flame retardant (IFR) and FeOOH were combined in accordance with their respective weight percentages, as indicated in Table 1. The IFR and FeOOH were blended using a shear mixer set to 1000 rpm for a period of 20 min. Subsequently, a styrene–acrylic emulsion was added to the aforementioned mixture and stirred until a uniform consistency was achieved at 400 rpm for 20 min. The coatings containing different wt% of IFR and FeOOH were applied to wooden boards of dimensions $100 \times 100 \times 3 \text{ mm}^3$ and $75 \times 75 \times 3 \text{ mm}^3$, which can be used for cone calorimeter and smoke density analyzer tests, respectively. The samples were then subjected to a 24 h curing period at room temperature. Following this, they were placed in a vacuum oven at 50 $^\circ\text{C}$ for a further 24 h. Table 1 shows the formulation of intumescent fire-retardant coatings.

Table 1. Formulations of intumescent fire-retardant coatings.

Sample Code	A0 (Blank Board)	A1	A2	A3	A4	A5	A6
Styrene–acrylic emulsion/wt%	0	21.0	21.0	21.0	21.0	21.0	21.0
IFR/wt%	0	49.0	48.5	48.0	47.0	45.0	41.0
FeOOH/wt%	0	0	0.5	1.0	2.0	4.0	8.0
Distilled water/wt%	0	20.0	20.0	20.0	20.0	20.0	20.0
Hydroxyethyl cellulose/wt%	0	7.0	7.0	7.0	7.0	7.0	7.0
Ethenediol/wt%	0	1.5	1.5	1.5	1.5	1.5	1.5
Antifoaming agent/wt%	0	1.0	1.0	1.0	1.0	1.0	1.0
Film-forming agent/wt%	0	0.5	0.5	0.5	0.5	0.5	0.5

2.3. Cone Calorimeter (CCT)

CCT was carried out by Stanton Redcroft in the UK in accordance with ISO 13943:2010 [21]. Each sample was $100 \times 100 \times 3 \text{ mm}^3$ in aluminum foil, placed horizontally in an external heat flux of 35 kW/m^2 .

2.4. Smoke Density Analyzer (SDA)

According to ISO 5659-2(2006) [22], smoke characteristics were measured by JQMY-2 smoke density tester (China Construction Bridge Corporation, Hong Kong, China). Each sample was $75 \times 75 \times 2.5 \text{ mm}^3$ in aluminum foil, placed horizontally in an external heat flux of 25 kW/m^2 , and lit or unlit separately to set up different control experiments.

2.5. Thermal Gravimetric Analysis (TGA)

Thermal gravimetric analysis was conducted under nitrogen flow on a DT-50 (Setaram, Caluire, France) instrument. Approximately 10.0 mg of the sample was placed in an alumina crucible and heated from 25 to 900°C . The heating rate was set at 10 K/min .

2.6. Scanning Electron Microscopy (SEM)

SEM observation was conducted using Hitachi X650 (Hitachi, Tokyo, Japan).

3. Results and Discussion

3.1. Cone Calorimeter Test

Cone calorimeters are focused via external radiation. The intensity of the fire is indicated by the heat release rate (HRR). However, fire hazards are often described using average or peak HRR (PHRR) [23,24]. Typically, for highly flame-retardant systems, the average HRR value is very low. PHRR values are used to indicate the intensity of a fire.

3.1.1. Heat Release Rate (HRR)

Figure 1 shows the HRR curves of various flame-retardant coatings obtained by CCT. Sample A0, without any flame-retardant coating, quickly ignited and reached a peak HRR (PHRR) value (256.3 kW/m^2) in about 100 s, with a total burn time of 350 s. The single peak shown is due to gradual burning. Compared with A0, the PHRR value of A1 (104.5 kW/m^2) of IFR alone is significantly reduced, greatly extending the ignition time. This is because the residual charcoal on the surface of the sample effectively delays the combustion process. The data show that MEL and APP are regarded as blowing agents and acid sources, respectively, while PER is regarded as a carbon donor. The carbon layer prevents heat from being transferred to the material below and prevents combustible gases from entering the flame zone. When FeOOH is added to the non-expandable flame-retardant coating system, the PHRR is lower than those of A0 and A1, containing only IFR. The order of the first PHRR value of each sample is $A0 > A1 > A5 > A6 > A3 > A2 > A4$. The samples designed for A4, including IFR and FeOOH concentrations of 2 wt%, had the lowest PHRR values (42.1 kW/m^2). It has been reported in previous studies [25,26] that the PHRR of samples with added FeOOH was significantly reduced, possibly due to the improved densification and weight of the carbon slag in cone calorimeter tests. This shows

that adding an appropriate amount of FeOOH to the coatings containing IFR can improve flame retardancy.

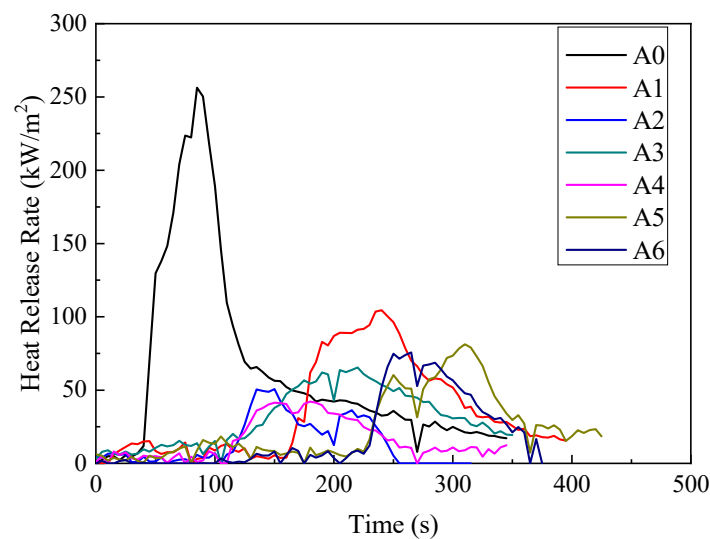


Figure 1. HRR curves of intumescent fire-retardant coatings at a flux of 35 kW/m^2 .

3.1.2. Mass

Figure 2 shows the weight of the intumescent flame-retardant coating's carbon residue. The carbon residue of samples with different FeOOH and IFR contents is greater than that of samples A1 containing only IFR and A0, representing a wooden board. When extended to 200 s, the carbon residue from sample A1 to sample A6 remained above 50%. When the combustion time reached 250 s, the FeOOH content in the coke residue of sample A4 was 2%, the highest of all the samples. This indicates that the optimal amount of FeOOH can promote the formation of carbon slag in the coating system. The cross-linking reaction between the metal oxide and the decomposing polymer may contribute. In addition, FeOOH causes a dense carbon residue to form on the coating surface during combustion, thus protecting the coating. The char residue impedes oxygen diffusion to the substrate and reduces the combustion rate of volatile gases, especially the aromatic gases that produce smoke [27,28].

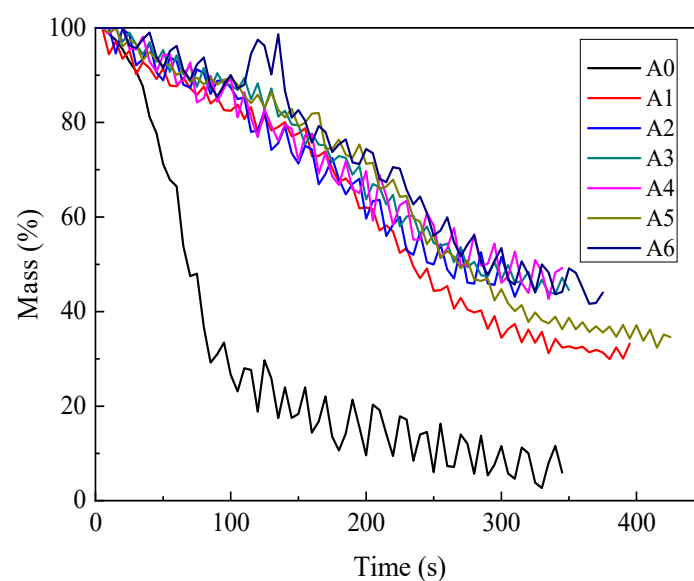


Figure 2. Mass curves of intumescent fire-retardant coatings at a flux of 35 kW/m^2 .

3.1.3. Total Heat Release (THR)

Figure 3 shows the THR for the samples. The THR value for sample A0 without flame retardants was significantly higher than that of the samples containing IFR or IFR/FeOOH flame retardants. It has been reported that the slope of the THR curve can be considered to represent flame spread [29,30]. Compared to A0, the THR curve slope of sample A1 containing only IFR was significantly reduced, indicating a slower rate of flame spread. In addition, when FeOOH is added to the crimp-type flame-retardant coating system, the THR is further reduced compared to sample A1 containing only IFR. This is due to the formation of a dense coke residue on the sample surface, which acts as a barrier and again limits the spread of the flame. Sample A4 had the lowest THR, at 290 s, of all the samples, which correlates with the HRR curve described in Figure 1.

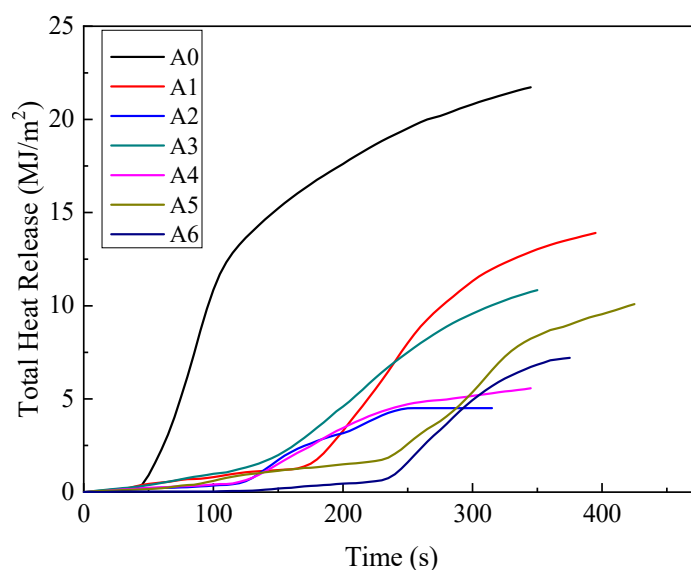


Figure 3. THR curves of intumescent fire-retardant coatings at a flux of 35 kW/m^2 .

3.1.4. Smoke Production Rate (SPR)

The term “smoke” is defined as a mixture of fuel gases, droplets, and solid particles in the air produced during combustion [15]. Several undesirable properties of smoke have been identified, above all, opacity and toxicity. It is important to know the dangers of smoking. The smoke production rate (SPR) is also important to estimate flame retardancy. The higher the SPR, the lower the flame retardancy of the material [31–34].

Figure 4 illustrates the SPR of intumescent fire-retardant coatings with varying quantities of FeOOH at a heat flux intensity of 35 kW/m^2 . The initial SPR peak ($0.0275 \text{ m}^2/\text{s}$) of sample A0, which lacks any fire-retardant coating, is higher than the other samples. The initial SPR peak in sample A1, comprising solely IFR, is approximately $0.007 \text{ m}^2/\text{s}$. The initial peak value of the samples exhibits a notable decline when FeOOH is introduced into the system. Upon exceeding a combustion time of 50 s, the SPR curves of A2, A3, and A4 exhibit a flattening tendency, due to the dense carbon layer on the surface of the samples. Furthermore, it can be observed that sample A4, which contains 2 wt% FeOOH, produces less smoke than the other samples throughout the entire combustion period. This finding aligns with the aforementioned results. However, the addition of FeOOH at 4 wt% and 8 wt% resulted in the production of more smoke than in other samples, even resulting in a second peak in SPR. This indicates that only an appropriate amount of FeOOH in the coating can ensure the smoke suppression performance.

The observed phenomenon is attributed to the fact that FeOOH promotes the carbonization process and the carbon layer. Furthermore, the formation of the carbon layer may result in a decrease in the smoke production rate. The protection provided by the

carbon layer may lead to a rapid reduction in the concentration of combustible gases and smoke-forming materials in the gas phase during combustion.

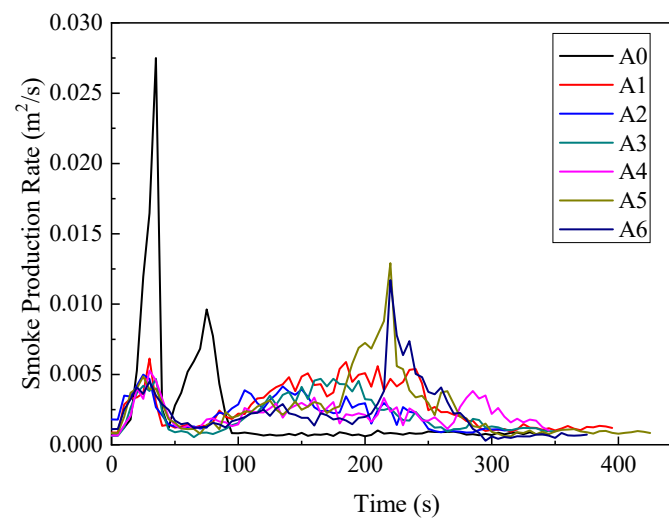


Figure 4. SPR curves of intumescent fire-retardant coatings at a flux of 35 kW/m².

3.1.5. Total Smoke Release (TSR)

TSR is employed to assess the impact of FeOOH on smoke reduction in the coating system. The TSR curves of intumescent fire-retardant coatings with a varying wt% of FeOOH are depicted in Figure 5. TSR represents the cumulative amount of smoke over a 4.0 min period and characterizes the total amount of smoke produced in a fully developed fire. Therefore, TSR is an effective indicator for determining the evacuation time and implementing safety procedures. In addition, the derivative of the evolution curve with respect to time is a valid indicator of the evacuation time and general safety procedures. Figure 5 illustrates that the TSR of sample A0, which lacks a fire-retardant coating, is higher than that of the other samples prior to 200 s. When the combustion time exceeds 200 s, sample A1, which contains only IFR, produces the greatest quantity of smoke among all samples. The addition of FeOOH has been observed to significantly reduce TSR. Sample A4 produces the least smoke of all the coatings. However, the addition of FeOOH at 4 wt% and 8 wt%, respectively, resulted in the generation of more smoke than the other samples. This suggests that the optimal amount of FeOOH in the coating is necessary for the smoke suppression performance, which aligns with the SPR results.

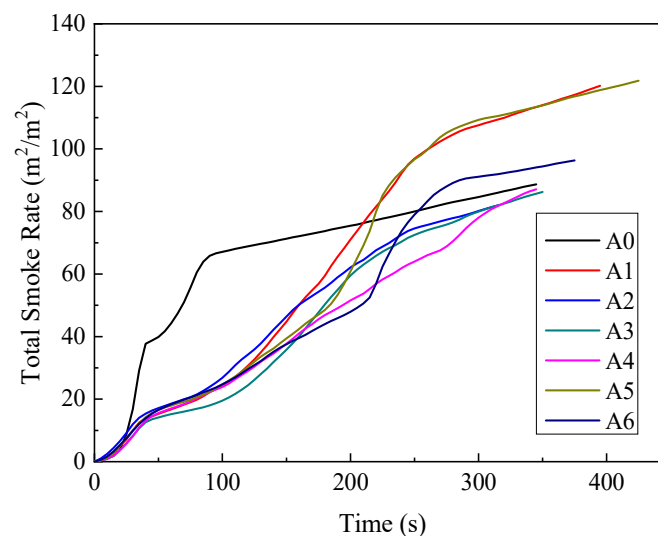


Figure 5. TSR curves of intumescent fire-retardant coatings at a flux of 35 kW/m².

The observed phenomenon can be attributed to the smoke suppression properties of FeOOH. The smoke suppression effect of FeOOH can be attributed to two mechanisms: the promotion of soot particle oxidation to CO and CO₂ and the formation of compact char residue, which prevents the diffusion of combustible gases into the air. Consequently, the objective of smoke suppression can be achieved [3,35].

3.1.6. Fire Performance Index (FPI) and Fire Growth Index (FGI)

FPI (m² s/kW) is the ratio of ignition time (TTI) to PHRR, and FGI (kW/m² s) is the ratio of peak HRR to time to PHRR (TTP) [22]. FPI and FGI are derived from cone calorimeter test results to reflect the safety level of the sample. The higher the security level, the higher the FPI value and the lower the FGI value [36,37]. Figures 6 and 7 show the FPI and FGI values, respectively. With the addition of FeOOH, FPI values increased significantly. In addition, the FeOOH content in sample A4 is 2 wt%, which has the lowest FGI and a relatively high safety level.

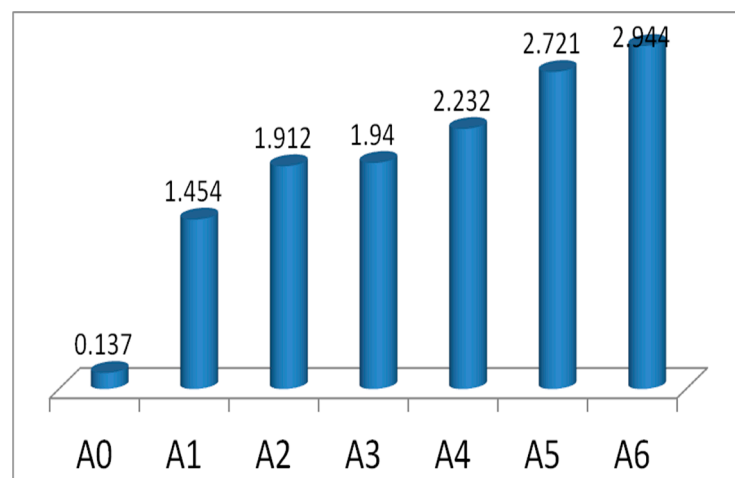


Figure 6. Fire performance index for intumescent fire-retardant coatings at a flux of 35 kW/m².

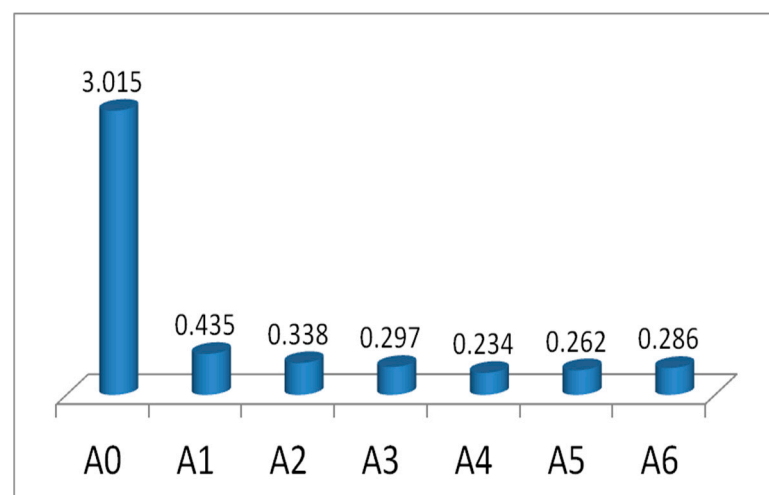


Figure 7. Fire growth index for intumescent fire-retardant coatings at a flux of 35 kW/m².

3.1.7. Morphology of Residues

Figure 8 shows the carbon residue's morphology produced by the expandable flame-retardant coating after testing with a cone calorimeter. An effective carbon layer is formed, preventing further combustion of the underlying material and slowing the thermal decomposition of the polymer. Among them, sample A0 has not been treated with a flame-retardant coating, and its residual carbon is the lightest and loosest. Sample A1 was coated

with IFR only, and its residual carbon content was higher. However, the surface of A1 residual charcoal is not smooth, and the density is not high. Samples with both IFR and FeOOH (A2 to A6) possess higher and regular carbon residues. With the increase in FeOOH content, the surface of the residual carbon becomes denser and denser. As the FeOOH content increases to 2 wt%, 4 wt%, and 8 wt%, the surface becomes increasingly smooth and compact. This indicates that FeOOH in the coating containing IFR contributes to the calcination of the carbon slag and produces excellent flame-retardant properties. Samples A5 and A6 produced more smoke and even produced a second smoke release rate peak when the FeOOH content reached 4% and 8%, respectively. It is concluded that only the FeOOH containing a proper mass fraction can produce good flame-retardancy and smoke suppression behavior in this system. The reason for this phenomenon may be that the addition of FeOOH is conducive to promoting carbonization, forming a carbon layer, and in the process of carbon layer formation, the smoke production rate may reduce. Due to the protection of the carbon layer, the formation of combustible gases and fumes in the gas phase during combustion is rapidly reduced.

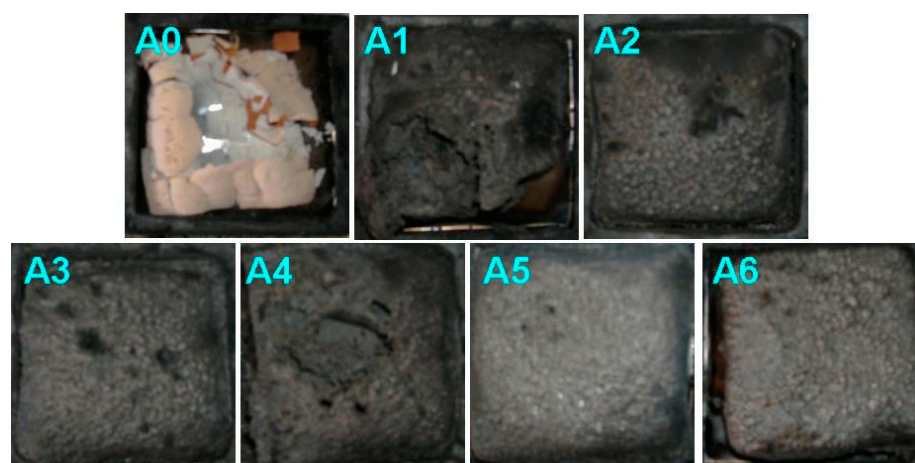


Figure 8. Char residues of intumescent fire-retardant coatings after CCT test.

When the increase in FeOOH rose to 4% and 8%, samples A5 and A6 produced more smoke than the other samples. This also means that only an appropriate weight fraction of FeOOH in this paint can produce better smoke suppression properties, which is consistent with the smoke rate results. The smoke suppression properties of FeOOH can explain this phenomenon. The smoke suppression effect of FeOOH has two aspects: on the one hand, it can promote the oxidation of CO and CO₂ by soot particles, and on the other hand, it promotes the formation of compact carbon residues to prevent the diffusion of combustible gases into the air. Therefore, it can achieve the purpose of smoke suppression.

3.2. Smoke Density Analyzer Test

Specific Optical Density (SOD)

Figure 9 depicts the specific optical density (SOD) curves for intumescent fire-retardant coatings containing varying quantities of FeOOH at 25 kW/m². The specific optical density (SOD) can be employed as a means of evaluating the quantity of smoke produced. It can be observed that the specific optical density of sample A1, which contains only IFR, is consistently lower than that of samples A2 to A6, which contain both IFR and FeOOH. This suggests that sample A1 produces a greater quantity of smoke than the samples containing FeOOH when the pilot flame is employed.

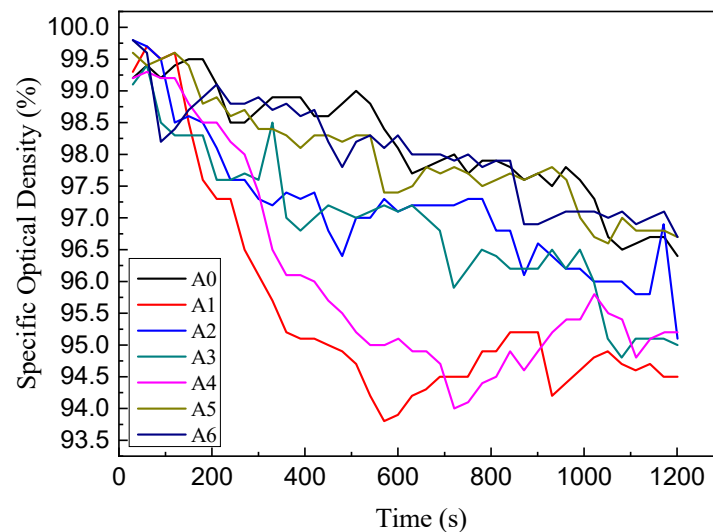


Figure 9. SOD curves of intumescent fire-retardant coatings at a flux of 25 kW/m^2 with flame.

Without the pilot flame, as illustrated in Figure 10, the incorporation of FeOOH was observed to result in a reduction in smoke production within these coatings. The specific optical density of sample A1, which contains only IFR, is lower than that of samples A2, A3, and A4, which contain FeOOH. However, when the concentration of FeOOH is increased to 4 wt% and 8 wt%, the specific optical density (SOD) of A1 is found to be significantly higher than those of A5 and A6. This suggests that the addition of an optimal quantity FeOOH to the coating can reduce the production of smoke. It can be posited that the optimal quantity of FeOOH promotes the char residues, resulting in the conversion of polymer carbon into smoke.

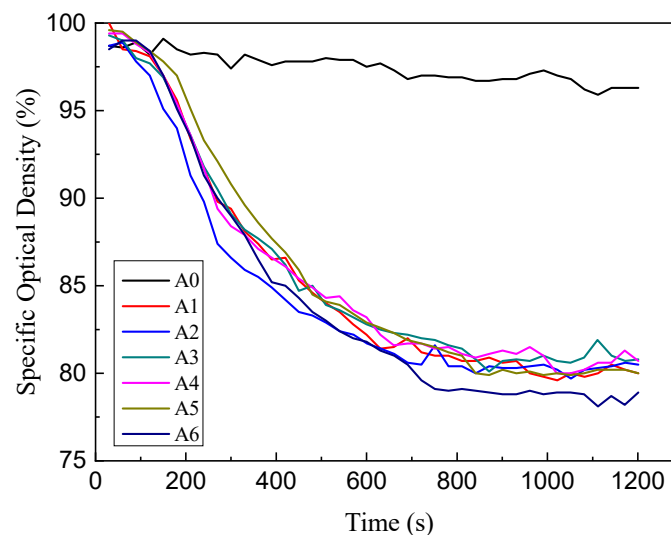


Figure 10. SOD curves of intumescent fire-retardant coatings at a flux of 25 kW/m^2 without flame.

The SDA results demonstrate that an optimal quantity of FeOOH can elicit a smoke suppression performance in this coating system, which corroborates with the cone calorimeter test outcomes. FeOOH diminishes the availability of carbon for the formation of aromatic compounds, which are the precursors in the reactions that result in the production of smoke and soot. Therefore, the transition of combustion products such as tar and soot particles to the gas phase is hindered, and the smoke density decreases greatly.

3.3. Thermal–Gravimetric Analysis Test

TGA represents an established methodology for the expeditious assessment of the thermal stability of a range of materials. In addition, the decomposition of the polymer at different temperatures can also be expressed according to the initial degradation temperature ($T_{5.0\%}$), the maximum degradation rate temperature ($T_{\max}$), and the carbon residue [27,31]. When the initial degradation temperature is 5.0 wt%, the mass loss of polymer is 5.0 wt%.

Figure 11 illustrates the TGA curves for intumescent fire-retardant coatings comprising varying quantities of FeOOH. It can be observed that sample A1 begins to decompose at approximately 250 °C (5.0 wt% weight loss). The char residue of A1 is consistently lower than that of samples A2 to A6, despite the varying FeOOH and IFR contents. This is illustrated in Figure 11. It can be observed that A1, which contains only IFR, is less stable than the other samples in the temperature range between 250 °C and 450 °C. Upon reaching a temperature of 450 °C, the char residue of samples A2–A6 with FeOOH was found to be greater than that of A1 with only IFR. The addition of FeOOH to the intumescent fire-retardant coating system enhances the carbon residues. This is in accordance with the above results.

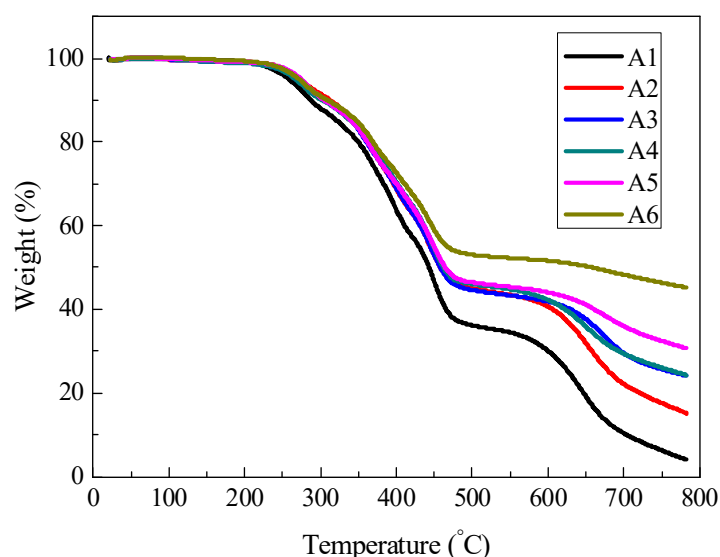


Figure 11. TGA curves of intumescent fire-retardant coatings.

It can be observed that intumescent fire-retardant coatings undergo four distinct stages of thermal degradation. The initial degradation, which occurs within the temperature range of 200–300 °C, is attributed to the decomposition of acrylic resin within the styrene–acrylic emulsion. Additionally, it can be demonstrated that the incorporation of FeOOH serves to accelerate the thermal degradation observed in the initial stage. The second and third degradations, occurring at 300–500 °C, are attributed to the degradation of melamine and APP, accompanied by the release of N_2 and NH_3 , and the decomposition of PER and MEL [37]. The incorporation of FeOOH was observed to enhance the thermal stability of these coatings during the second and third degradations, a finding that aligns with the TGA results. The fourth degradation, occurring at 500–800 °C, was due to the decomposition of amorphous carbon formed in the preceding stage. The maximum mass loss rate ($T_{\max}$) temperature of the A1 sample was about 650 °C, which was lower than the other samples degraded by FeOOH for the fourth time; thus, FeOOH could improve the thermal stability of the coating.

3.4. Scanning Electron Microscopy (SEM)

In order to further understand the link between the structure of the expanded charcoal and its refractory properties, scanning electron microscopy (SEM) was carried out on

the charcoal residue remaining after the cone calorimeter test. Figure 12 shows the SEM photographs of the expanded char of samples A1 and A4, magnified 300 and 2000 times, respectively. The surface of sample A1, which contains only IFR, shows many holes and cracks, while the carbon layer has an irregular appearance. In contrast, the surface of A4 carbon slag, which contains both IFR and FeOOH, is relatively smooth and has an emulsion structure. This may be due to FeOOH's ability to promote charring and the tight carbon layer, limiting heat release and smoke generation.

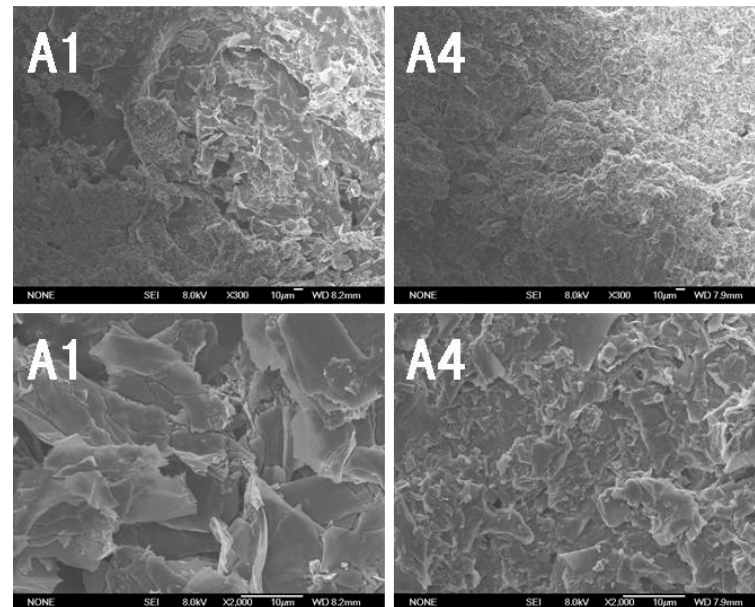


Figure 12. SEM of char residue from sample A1 and sample A3 after CCT.

4. Conclusions

A range of intumescent flame-retardant coatings (IFR, based on ammonium polyphosphate (APP), pentaerythritol (PER), melamine (MEL)), styrene–acrylic emulsion coatings, and iron oxide yellow (FeOOH) coatings were prepared.

- (1) FeOOH can significantly reduce the HRR, THR, SPR, and TSR of paint samples, improve the quality of the carbon residue, reduce the smoke production of the samples, and change the structure of the carbonization residue;
- (2) The weight of char residue increases with the increase in the addition of FeOOH;
- (3) Samples containing 2% FeOOH/IFR have a greater effect on the fire- and smoke suppression properties of fire-retardant paint samples.

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