Influences of flame-retardant fillers on fire protection and mechanical properties of intumescent coatings

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ABSTRACT

A combination of acrylic binder and flame-retardant ingredients was used to synthesize the solvent-borne intumescent coatings designed for steel substrates. The influences of individual and various combinations of flame-retardant fillers on the fire protection and mechanical properties of the coatings were characterized by using Bunsen burner, thermogravimetric analysis, limiting oxygen index, field emission scanning electron microscopy, freeze–thaw cycles, static immersion and pull-off type equipment. It was found that the combination of aluminium hydroxide ($Al(OH)_3$) and titanium dioxide (TiO_2) has significantly improved the fire protection, thermal stability and water resistance of the coating. This formulation had an LOI value of 34, which indicated good flammability resistance of the coating. The adhesion strength tests showed that the coating added with magnesium hydroxide ($Mg(OH)_2$) exhibited maximum bonding strength to the metal surface due to its effective interface adhesion. Hence, the findings from this study revealed that the selection of appropriate combinations of flame-retardant fillers strongly influenced the physical and chemical properties of the coatings.

1. Introduction

Application of intumescent coating in buildings has recently become one of the most popular passive fire protections (PFP) to meet the requirements of legislation and to prevent failure of major building components in fires. Structural fire safety is one of the key considerations in the design of built infrastructure as it could save precious human lives and assets. Intumescent coatings are not as frequently used as PFP due to lack of knowledge and exposure to its potential as highly effective fire-resistive components. PFP of steel members is usually achieved by using materials such as cement-based sprays, boards, batt materials and intumescent coatings [1]. This project is aimed at investigating the fire-resistant properties of intumescent coatings on steel, which takes into account the qualitative effect of an endothermic metamorphic reaction of the flame-retardant filler.

The intumescent coating is designed to insulate the steel substrate. It prevents the temperature of the steel from rising to a critical point of 550 °C and maintains the steel's integrity for 1–3 h in the event of a fire. These coatings have widespread use as PFP to the structural steel in modern architectural designs, whilst at the same time maintaining an aesthetically pleasing appearance. Recently, many researchers have extensively studied the performance of intumescent coatings in terms of fire protection, water resistance, thermal stability and mechanical properties [2–12]. When exposed to irradiation, intumescent coatings decompose into voluminous, multi-cellular char layers with thermal insulation properties sufficient to protect the underlying material from reaching softening or pyrolysis temperature.

The commonly found halogen flame-retardants have been shown to leach out of polymers into the natural environment, where their presence is now ubiquitous, and some are proven endocrine disruptors [13,14]. These problems have driven the search for alternative "halogen-free" fire retardant components, which include metal hydroxide [15,16] and carbonate fillers [17], phosphorus compounds [18] as well as a range of more esoteric materials, such as clay and silica nanoparticles [19,20], carbon nanotubes [21], expandable graphite [22] and metal chelates [23,24]. Some of these fire-retardant materials, such as the volatile phosphorus compounds, act like halogens, which inhibit the gas phase combustion reactions, while the majority creates an insulating layer resulting from the buildup of char or intumescence in the attempt to stabilize the condensed phase through barrier

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formation. Incorporation of any non-combustible filler will slow the rate of burning and reduce the flammability of a polymer. There may also be synergistic or antagonistic catalytic [25] or other surface effects associated with the filler, which changes the melt rheology of the polymer, thereby affecting its tendency to drip [26].

Moreover, certain inorganic materials decompose endothermically with the release of inert gases or vapour, enhancing the potential fire retardant effect. In order to be effective, the decomposition must occur in a narrow window above the polymer's processing temperature, but at or below its decomposition temperature. In practice, most of the suitable materials used as fire retardant fillers for intumescent coatings are carbonates or hydroxides from groups II and III of the periodic table. They have three fire retardant effects, in addition to those of the inert fillers described above.

(1) Endothermic decomposition, absorbing heat and therefore keeping the surrounding polymer cooler.
(2) Production of inert diluent gases. Flaming reactions require a critical concentration of free radicals to be self-sustaining. If this concentration falls sufficiently, for example by the release of water or carbon dioxide, flame extinction will occur.
(3) Accumulation of an inert layer on the surface of the decomposing polymer, shielding it from incoming radiation, and acting as a barrier to oxygen reaching the fuel. It also prevents flammable pyrolysis products from reaching the gas phase, and radiant heat from reaching the polymer.

This work presents an experimental study to assess the efficiency of solvent-borne intumescent coatings on steel. The influence of individual and combinations of flame-retardant fillers on fire protection performance, thermal degradation, water resistance and mechanical properties of the intumescent coatings were studied by using thermogravimetric analysis (TGA), Bunsen burner test, limiting oxygen index test (LOI) and Instron Micro Tester equipment. Field emission scanning electron microscopy (FESEM) was used to observe the surface morphologies of the coatings and char layers. The paper comparatively analyzes water resistance of the flame-retardant intumescent coatings.

2. Experimental work

2.1. Materials

In this research, acrylic resin with a softening point of about 180 °C was used as a binder. Acrylic resin is a 100% solid thermoplastic substance derived from acrylic acid, methacrylic acid, esters of these acids, or acrylonitrile and is a general-purpose polymer with excellent hardness, broad compatibility and good weather-resistance. Acrylic resin in the form of acrylic copolymer (M_w = 60,000) was purchased from Mitsubishi Rayon Co., Ltd. (Tokyo, Japan).

Three main flame-retardant agents and three fillers were chosen for this study:

- A commercial crystal phase II ammonium polyphosphate (APP) (n > 1000) derivative (Fig. 1) which acts as an acid source.
- Melamine (MEL) which acts as a blowing agent.

- Pentaerythritol (PER) which acts as a carbon source.
- Magnesium hydroxide ($Mg(OH)_2$) and aluminium hydroxide ($Al(OH)_3$) which act as flame-retardant fillers.
- Titanium dioxide (TiO_2) which acts as a pigment as well as non-combustible filler.

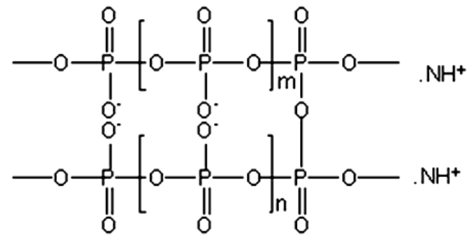

Fig. 1. Ammonium polyphosphate phase II.

The compositions of the intumescent coatings are listed in Table 1. The formulations were prepared by mixing the components using a high-speed disperse mixer. The prepared coatings were coated on steel plates using a gun sprayer.

2.2. Fire protection performance test

This test was used to characterize the formation of char and reaction of the intumescent coating, and to compare the evolution of temperature on the single-side coated steel plates when exposed to fire. The prepared coating was applied onto grit blasted steel plates (dimensions: 100 mm × 100 mm × 2.6 mm) and allowed to dry at room temperature. This process was repeated 6–8 times until a 1.5 ± 0.2 mm dry film thickness was obtained. Then, a high-temperature flame (about 1000 °C) was applied with a Bunsen burner for 60 min to the vertically mounted coated plate. The gas consumption of the Bunsen burner was at a rate of 160 g/h and the distance between the fire source and sample was about 20 mm (Fig. 2).

Measurement of the temperature profile during exposure to fire was recorded using a digital handheld thermometer. In this experimental work, 400 °C was chosen as the critical temperature for steel to ensure a higher level of safety (the standard critical temperature is 550 °C) [3].

2.3. Limiting oxygen index (LOI)

In the LOI test, a flat steel bar (dimensions: 127 mm × 27 mm × 3.1 mm) coated with intumescent coating (thickness of

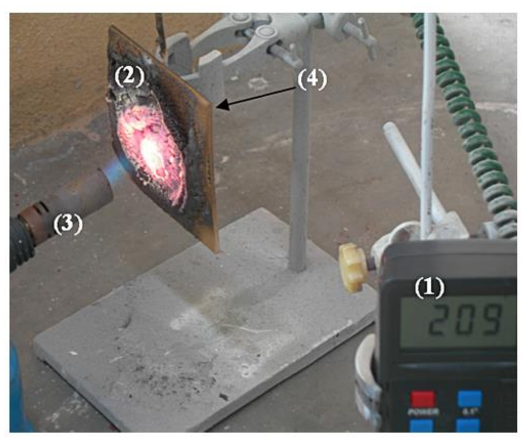

Fig. 2. Set-up for the fire protection performance test: (1) digital thermometer; (2) test sample, (3) Bunsen burner and (4) thermocouple.

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