



Effects of ammonium polyphosphate and boric acid on the thermal degradation of an intumescent fire retardant coating



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ARTICLE INFO

Keywords:

Intumescent fire retardant coating
Expandable graphite
Tga
XPS analysis
Weathering test

ABSTRACT

An intumescent coating is an insulating system designed to decrease the heat transfer a substrate structure. The intumescent fire retardant (IFR) coating presented here is based on expandable graphite (EG), ammonium polyphosphate (APP), melamine, and boric acid. Bisphenol epoxy resin BE-188 (BPA) was used as a binder with ACR Hardener H-2310 polyamide amine. Different formulations were developed to study the effects of APP and boric acid on char expansion, heat shielding, char morphology and char composition after a fire test. The coating was tested at 950°C for one hour. Char expansion was examined by furnace using a fire test. The results show that the coating is stable on the substrate. The morphology of the char was studied using Field Emission Scanning Electron Microscope (FESEM) of the coating after a fire test. X-ray Diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) showed the presence of carbon, borophosphate; boron oxide and sassolite in the residual char. Thermogravimetric analysis (TGA) including derivative plots showed that boric acid and APP enhance the residual weight of intumescent fire retardant coating. X-ray photoelectron spectroscopy (XPS) confirmed that IF5 recorded better carbon content up to 47.45 wt%, in the residual char that enhanced the fire resistance performance of the coating. An accelerated weathering test according to ASTM D 6695-03 showed that the IF5 coating continued its reliability up to 90 days in the hastened weathering chamber.

1. Introduction

Passive fireproofing materials composed of insulating systems (forming a carbonaceous char on the substrate on ignition) are commonly used to protect steel structures. These materials are designed to decrease heat transfer from a fire to the structure being protected. These systems are usually mineral-based or organic resin-based products, known as intumescent coatings. These coatings are designed to perform under severe conditions to maintain the steel integrity between 1–3 h when the temperature of the surrounding is in excess of 1100°C. This duration is considered sufficient for evacuation of personnel and to control the fire [1–3]. Previous studies have shown that structural steel maintains its integrity up to 500°C. Above this temperature steel structures collapse within 45 min [3–6]. Therefore, to ensure the protection of steel structures, it is essential to maintain its temperature below 500°C during fire incidents.

Ammonium phosphate was first recommended as a flame retardant in theater curtains by Gay-Lussac in 1821 [7]. Mono and diammonium phosphate, or a mixture of the two, are widely used as flame retardants

in a broad range of cellulosic materials such as paper, cotton, and wood [8]. The flame retardant formulations based on these salts are generally nondurable, due to their water solubility making them readily susceptible to being leached from the material matrix. Ammonium polyphosphate (APP), on the other hand, is a moderately water-insoluble, high melting solid with high phosphorous content (up to 30%). APP exists in several crystalline forms and the commercial products differ in molecular weights, particle size, and solubilities. These salts are used in intumescent paints with pentaerythritol as the carbon source and melamine as the blowing agent [9]. The intumescent coatings normally contain a resinous binder, an acid source, gas source and other inorganic fillers [10–12]. Phosphorus is known to promote char formation, forming a protective coating. Phosphoric acids form coatings and protect the substrate similar to borate glasses which form when boric acid and borax are used [13]. The incorporation of large quantities of a filler will dilute the polymer and thereby reduce the rate of formation of decomposition gases.

Boron compounds are also widely used as a flame retardants and compounds such as borax, boric acid, zinc borate, barium metaborate,

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Table 1
The mass % of the ingredients used to develop ICFs.

Formulation	EG	APP	MEL	Boric Acid	Epoxy	Hardener
IF1	5.8	0	11.76	11.76	47.12	23.56
IF2	5.8	11.76	11.76	0	47.12	23.56
IF3	5.8	11.76	11.76	5.55	43.42	21.71
IF4	5.8	8.76	11.76	8.55	43.42	21.71
IF5	5.8	5.76	11.76	11.55	43.42	21.71

and ammonium fluoroborate are well-known fire retardants [14]. Boron compounds act in both the condensed and vapor phase as fire suppressants. Most boron complexes are Lewis acids, which promote crosslinking of polymeric material on thermal degradation and thus minimize polymer decomposition and the evolution of volatile combustibles. Boron compounds can also react with a hydroxyl group in the polymer to form a glassy ester. This ester forms a char on the substrate surface and reduces solid-state carbon oxidation by protecting the underlying material [15].

Boric acid inhibits the release of combustible gases from burning cellulosic materials, such as cotton, wood, and paper-based products. Boric acid also releases chemically bonded water to dilute the concentration of pyrolytic fuel fragments and a carbon char is formed that further inhibits combustion [16,17]. It is used as a flame retardant in plastics, textiles, coatings, and there are also other industrial products that contain boric acid to strengthen their ability to withstand exposure to flames [18–20]. Similar to the phosphoric acids resulting from phosphate esters, boric acid dehydrates oxygen-containing polymers yielding char. The glassy coating and the char protect the substrate from oxygen and heat. It has been reported that boric acid impressively enhances the thermal stability of ethylene-vinyl acetate (EVA) at temperatures below 350 °C; but, it promotes char formation at higher temperatures, perhaps due to the presence of the heat resistant B_2O_3 [21].

The objective of this research work was to investigate the effects of APP and boric acid on the heat shielding of coating, char expansion, char morphology, char composition and thermal degradation and residual weight of expandable graphite based intumescent coating formulations (ICFs). Finally, the performance in terms of char expansion, and char morphology after an accelerated weathering test was also explored.

2. Experimental

2.1. Materials and methods

Flake graphite, melamine, and boric acid were purchased from

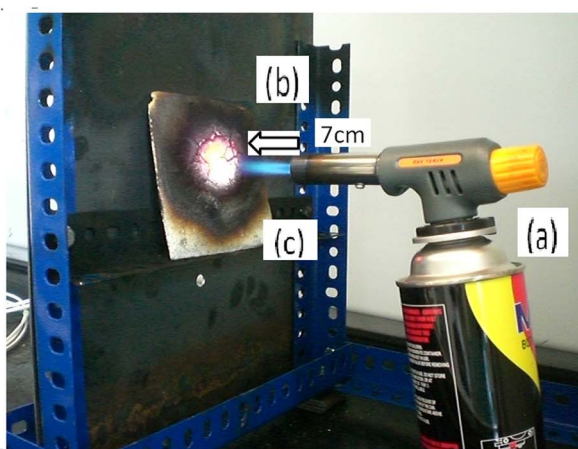


Fig. 1. Fire test setup for the heat insulation test of the intumescent formulation: (a) portable Bunsen burner, (b) stand, and (c) sample of intumescent coating [15].

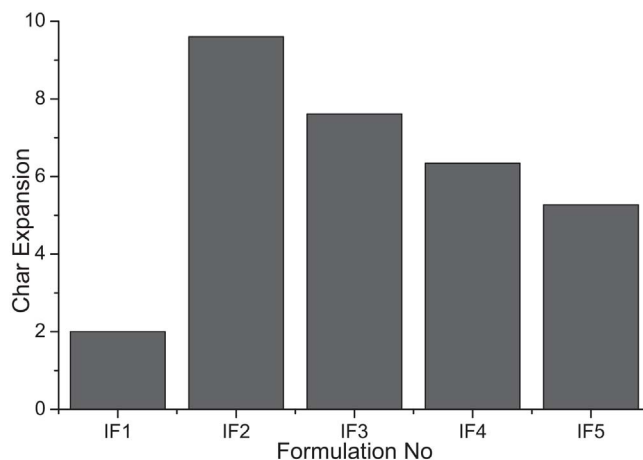


Fig. 2. Effect of boric acid and APP on char expansion of ICFs.

Sigma-Aldrich (M) Sdn Bhd. Malaysia. Ammonium polyphosphate was provided by Clariant (Malaysia) Sdn Bhd. Acetic acid, sulphuric acid, $KMnO_4$, bisphenol An epoxy resin BE-188 (BPA) and ACR Hardener H-2310 polyamide amine were bought from Mc-Growth chemical Sdn Bhd. Malaysia. Structural steel A36 M was supplied by TSA industries (Ipoh) Sdn. Bhd. Malaysia.

2.2. Coating preparation

Expandable graphite was prepared by treating of flake graphite with acetic acid, sulphuric acid and potassium permanganate using a ratio of 1:2:0.5:0.07, respectively [15,22]. All intumescent ingredients were mixed in the mass percentage as given in Table 1. A shear mixer was used for the mixing of the coating at 40 rpm for 30 min. A structural steel plate of area $10 \times 10 \text{ cm}^2$ was used as a substrate. The coating was applied to the steel substrate using a brush. The thickness of the coating was maintained at 1.5 mm and measured using a digital vernier caliper. The coated substrate was cured in an oven at 60 °C for one hour. Five formulations prepared to study the effects of boric acid and APP on heat shielding and char expansion for intumescent coating formulations (ICFs) are listed in Table 1. The formulations were further characterized by XRD, FTIR, SEM, and TGA.

3. CHARACTERIZATION OF ICFs

3.1. Heat shielding effect

A UL-94 fire test was conducted for each formulation to evaluate the

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