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Effects of zirconium silicate reinforcement on expandable graphite based intumescent fire retardant coating

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ABSTRACT

The effects of zirconium silicate as a fire retardant reinforcement in the mixture of expandable graphite (EG), ammonium poly phosphate (APP), melamine, boric acid, bisphenol A epoxy resin BE-188(BPA) and ACR Hardener H-2310 polyamide amine are presented. Different formulations were developed to study the effects of zirconium silicate on char expansion, heat shielding, char morphology and composition after fire test. The coatings were tested at 950 °C using Bunsen burner for 1 h. The results show state that the zirconium silicate enhanced fire protection performance of intumescent coating. The morphology of the char was studied by Field emission scanning electron microscope (FESEM) after furnace fire test. X-ray Diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) results showed the presence of graphite, borophosphate; boron oxide and boric acid in the char. Thermogravimetric analysis (TGA) showed that zirconium silicate enhanced residual weight of char. X-ray photoelectron spectroscopy (XPS) analysis showed that 5% zirconium silicate enhanced the carbon content up to 60.87% and lowered oxygen content to 28.09% in the residual char which proved helpful in improving the fire resistance performance of coating. Pyrolysis analysis confirmed that IF5-ZS releases less gaseous products concentration compared to IF-control coating.

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1. Introduction

The need for fireproofing material became important in the nineteenth century due to the commercialization of cellulose nitrate plastic [1]. A fire retardant system is a compound or compositions added to materials, which increases a given material's resistance to combustion [2]. Effective fire retardant needs to hinder the supply of one or more of the elements required for sustained combustion [3]. The objective of the fire retardants is to lower a polymers intrinsic fire hazard by decreasing the rates of burning and flame spreading under fire conditions. The use of the fire retardants may avoid a small fire from flattering a major disaster [4]. In order for a fire retardant to be effective, it must interact and interfere with the degradation of the host polymer at the polymer's degradation temperature. The degradation temperatures for the most widely used polymers are between 200 °C and 400 °C [5,6].

Different types of flame retardants, including intumescent systems with and without the additions of the fillers have been implemented with great success. Another new field of interest is the use of nano-clay composites as flame retardants [7] and reasonable flame retardancy can be achieved at very low amount, i.e. 5%. During the intumescent process, the binder becomes important due to two effects: it contributes to the char layer expansion and ensures the formation of uniform char foam structure [3–5].

Zirconium is used as filler in intumescent fire retardant coating (IFRC). The use of zirconia with borate as additives in organic polymers reduces their flammability. Zirconia is modified at high temperature and provides the effects of fire retardant. It is preferred to use powder form of zirconia in the intumescent coating for easy dispersion in the coating material [8]. In thermal barrier coating, $ZrSiO_4$ or zircon has been extensively investigated due to its chemical inertness and high temperature and thermal stability [9]. Zirconium Oxychloride used as a fire retardant in wool fabrics, alpha-zirconium phosphate used in flame retardancy and thermal properties of epoxy acrylate resin. Zirconium silicate is selected as a filler material in intumescent coating formulation was due to its high temperature resistance.

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The objective of this research work was to investigate the effects of zirconium silicate reinforcement on the heat shielding, char expansion, char morphology, char composition, residual weight and analysis of gaseous products of control formulation IF-control coating.

2. Materials and methods

Flake graphite, melamine (Mel) and boric acid (B.A) were purchased from Sigma–Aldrich (M) Sdn Bhd. Malaysia. Ammonium poly phosphate (APP) was provided by Clariant (Malaysia) Sdn Bhd. Acetic acid, sulphuric acid, $KMnO_4$, binder system bisphenol A epoxy resin BE-188 (BPA) and ACR Hardener H-2310 polyamide amine and Zirconium silicate (ZS) were purchased from Mc-Growth chemical Sdn Bhd. Malaysia. The Field emission scanning electron microscope (FESEM) micrograph of Zirconium silicate showed the particle size 22.27 μm in Fig. 1. Structural steel A36M was supplied by TSA industries (Ipoh) Sdn. Bhd. Malaysia.

2.1. Coating preparation

Expandable graphite (EG) was prepared by reacting flake graphite with acetic acid, sulphuric acid and potassium permanganate using ratio 1:2:0.5:0.07 respectively [10]. All intumescent ingredients were mixed with their respective weight percentage as stated in Table 1. The shear mixer was used for the mixing of ingredients with epoxy and hardener at 40 rpm for 30 min. The structural steel plate of area 100 cm^2 was used as a substrate. The coating was applied using brush on the steel substrate and an average thickness of coating was maintained at 1.5 mm and it was measured by digital vernier calliper. The coated substrate was cured in the oven at 60 °C for 1 h.

Table 1 showed six intumescent coating formulations (ICF) prepared to study the effects of zirconium silicate on heat shielding and char expansion of the IFRC. The char was characterized by FESEM, X-ray Diffraction (XRD), Fourier Transform Infrared spectroscopy (FTIR), and X-ray Photoelectron Spectroscopy (XPS) analysis. The residual weight was obtained using Thermogravimetric Analysis (TGA). The composition of gaseous product was determined by Pyrolysis Gas Chromatography Mass Spectroscopy (Py-GCMS).

Table 1
The weight% of zirconium silicate reinforced intumescent coating.

Formulation no	EG	APP	Mel	Boric acid	Zirconium silicate	Epoxy	Hardener
IF-Control	5.8	11.76	5.76	11.5	–	43.42	21.71
IF1-ZS	5.8	11.76	5.76	11.5	1.0	42.76	21.38
IF2-ZS	5.8	11.76	5.76	11.5	2.0	42.10	21.00
IF3-ZS	5.8	11.76	5.76	11.5	3.0	41.42	20.71
IF4-ZS	5.8	11.76	5.76	11.5	4.0	40.76	20.38
IF5-ZS	5.8	11.76	5.76	11.5	5.0	40.10	20.06

3. Characterization of intumescent coating and char

3.1. Fire test

The fire test was conducted for each coating formulation to evaluate the penetration of heat from fire to the steel substrate. A portable Bunsen burner was used to burn the coating on the steel substrate and the distance between the coated substrate and burner was maintained 7 cm. Three thermocouples type K were connected to Anarittsu Data logger, Input Channel 6 Model AM-8000K with Anarittsu software and other end of three thermocouples were connected to the back of coated substrate. The temperature of the steel plate was recorded for 60 min at an interval of 1 min. The butane gas was used during Bunsen burner test and temperature of flame was determined using K type thermocouple and temperature was recorded by Data Logger. The butane gas has maximum temperature of 1150 °C.

3.2. Furnace test

To analyze the physical properties of char such as char expansion and char structure after fire test. The intumescent coatings

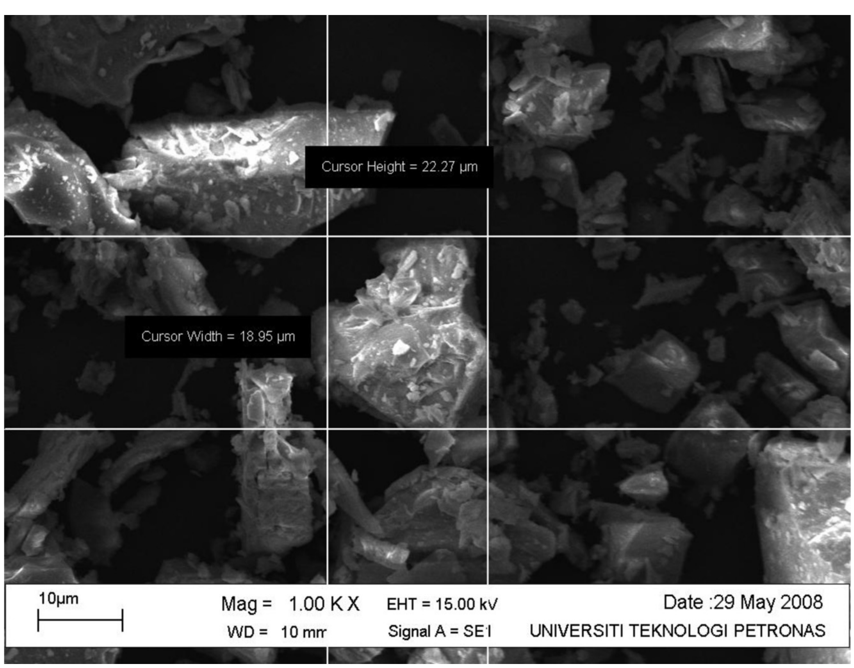

Fig. 1. FESEM image shows particle size of zirconium silicate used in the study.

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