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Thermal and pyrolysis analysis of minerals reinforced intumescent fire retardant coating

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ABSTRACT

This study presents the results of intumescent fire retardant coatings (IFRCs) developed to investigate the synergistic effects of mineral fillers (clay and wollastonite) based IFRC towards heat shielding, char expansion, morphology, composition, gaseous products and residual weight. The fire test has been performed to study the heat shielding effect of IFRCs on the substrate using UL-1709 standards. The results showed the synergistic effect of clay and wollastonite using 5 wt.% enhanced the fire protection, performance with recorded substrate temperature 113 °C after 1-h fire test. Field Emission Scanning Electron Microscopy (FESEM) and High-Resolution Transmission Electron Microscopy (HRTEM) showed the micrograph of compact char structure that increased char integrity due to the presence of inorganic fillers. X-ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) results showed the presence of boron phosphate, silicon phosphate oxide, aluminum borate in char that improved the thermal performance of IFRC up to 800 °C. X-Ray Photoelectron Spectroscopy (XPS) confirmed that 5 wt% (IFRC-5) of clay and wollastonite increased the carbon content up to 50.67%, lowering oxygen content to 27.73% in the char that enriched the fire resistance performance of the coating. Pyrolysis-Gas Chromatography-Mass Spectrometry (Pyrolysis GC–MS) confirmed that formulations IFRC-5 released less gaseous product concentration compared to IFRC-C and maximum reduction in gases was recorded from $3.4e^{+07}$ to $1.08e^{+07}$. Thermogravimetric analysis (TGA) demonstrated in residual weight was increased to 46.45% for IFRC-5 which resulted in the high thermal stability of the coating.

1. Introduction

In recent decades, the disastrous accident caused by damage to steel structure in the fire has reminded people of the risk of fire under the structural structure. During the fire, the temperature of structural steel rises to 800 °C in less than 10 min and steel starts losing its load bearing capability after 450 °C [1–5]. An official report on the 11th September tragedy found that the length of time the buildings remained upright after being hit by two passenger aircraft astonished most observers, including knowledgeable engineers. It claimed this resistance was a testament to its good construction and undoubtedly saved the lives of many within the building [6]. Therefore, the fire protection installed on the building was almost certainly superior to that which was required by the original design requirements that existed when the building was constructed. Some efficient protective ways, such as the application of flame retardant coatings can be used to protect steel structures from fire.

The intumescent coating layer has been implemented widely in structure as well as highly sealed steel structure as fire protection mechanism. As passive fire retardant, intumescent has been permanently attached to the material as a coating layer for a longer period of time and once activated when the fire or heat existed [7,8]. The intumescent coating will delay the heat transfer to the structure inside by forming a protective layer or char. This material forms an insulating barrier which protects the substrate from the rapid increase in temperature [9–15]. Such protection is useful for increasing the time to collapse under fire for steel structures or to upgrade fire resistance of walls or ceilings of various materials, avoiding diffusion of fire, smoke, and temperature on the opposite side of the wall. To improve the anti-oxidation or fire-resistant performance of the intumescent char, the synergistic effect of various fillers are giving significant attention to researchers [16–19].

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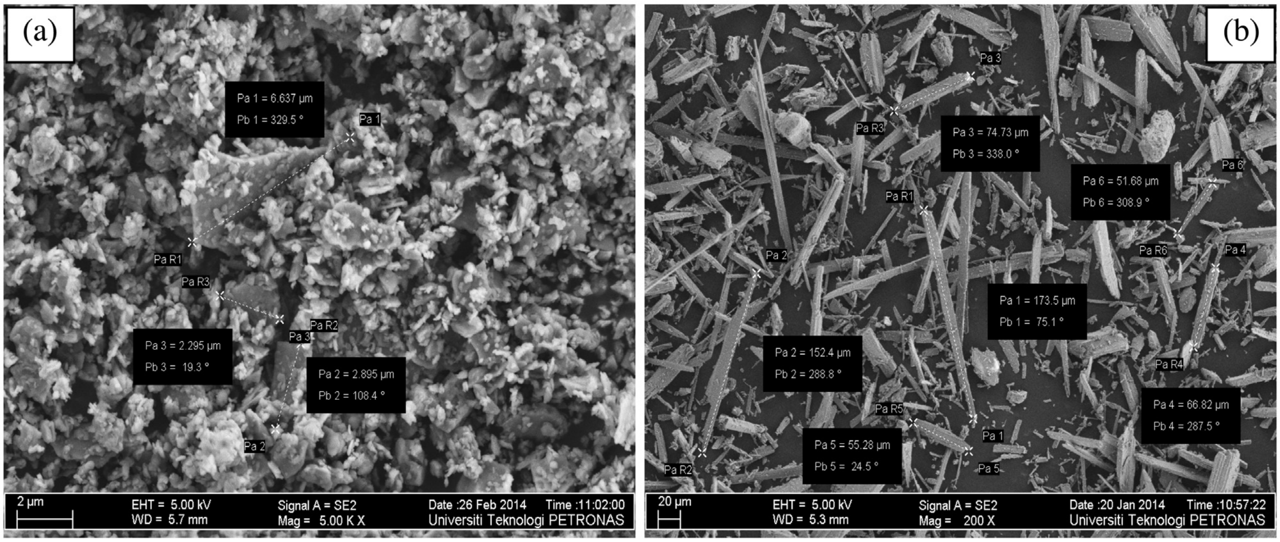

Fig. 1. Microstructure of kaolin clay (a) and wollastonite (b).

Table 1
Weight Percentage of ingredients of IFRC formulations developed in this study with additive ratio of clay and wollastonite (50:50).

No	*EG*	*APP*	*Mel*	*Boric Acid*	*Epoxy*	*Hardener*	*Clay: Wollastonite 50:50*
IFRC-C	5.8	11.76	5.76	11.5	43.42	21.71	0
IFRC-1	5.8	11.76	5.76	11.5	42.76	21.38	1
IFRC-2	5.8	11.76	5.76	11.5	42.1	21	2
IFRC-3	5.8	11.76	5.76	11.5	41.42	20.71	3
IFRC-4	5.8	11.76	5.76	11.5	40.76	20.38	4
IFRC-5	5.8	11.76	5.76	11.5	40.16	20.09	5

Many researchers have worked [20–27] on various coating formulations for fire retardant, however, the adhesion of the char with the substrate for these coating formulations at high temperature has a great challenge and these coatings are considered not effective to protect substrate. In order to resolve this problem, the concept of synergism is used to increase the adhesion of the char and to achieve enhanced thermal performance in composites and coatings [28–31]. Various coatings have been developed to investigate the effects of fillers on thermal performance of the intumescent coating. The addition of clay in APP-PER-PP formulations showed the good synergistic effect and improved the fire retardant performance of polypropylene [23]. Effect of boric acid with kaolin clay in IFRC resulted in the good thermal performance of intumescent coatings [32]. Inorganic filler alumina, alumina trihydrate and fumed silica enhanced the residual char weight and improved fire performance [26,33]. Synergies of nano clay and multi-walled carbon nanotubes in intumescent organ phosphorous flame retardants have enhanced the thermal stability of intumescent formulations [34,35].

The kaolin clay and wollastonite are stable at high temperature due to the presence of silicate structure. Kaolin is a fine clay mineral and has two layers of the crystal i.e. silicone-oxygen tetrahedral layer which are attached with alumina octahedral layer. Kaolinite is the main ingredient of kaolin whose chemical composition is $Al_2Si_2O_5(OH)_4$ (theoretically 39.8% + alumina 46.3% + silica 13.9%). Clay can be used in intumescent systems as flame retardant systems [36–38]. Wollastonite, also known as calcium metasilicate, is a naturally occurring mineral. It has been used usually in polymer composites as a thermal stabilizer and mechanical reinforcement. Some authors presented the efficiency of wollastonite as flame retardants in the silicone-based materials [39].

In this research work, the combination of kaolin with wollastonite was investigated as fire-retardant material in expandable graphite based intumescent coating. The equal ratios (50:50) and increasing the weight percentage (1–5) of kaolin and wollastonite have been incorporated in the intumescent coating, to achieve better char with an expanded structure that develops on exposure to

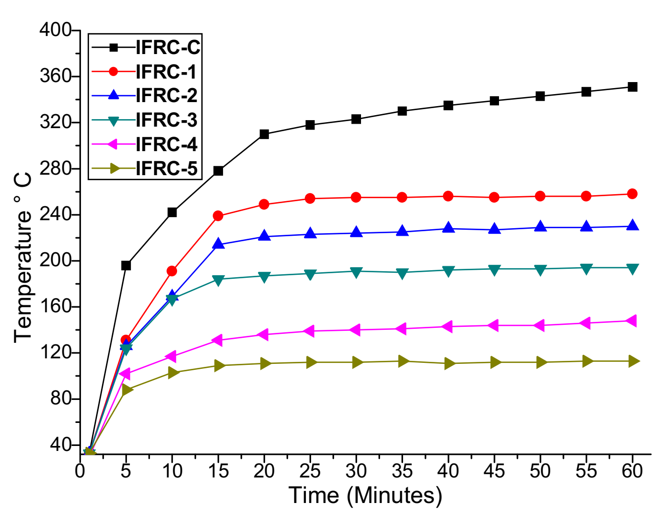

Fig. 2. Time and backside steel substrate temperature of IFRCs for 1 h fire test.

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