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Effect of basalt fibers dispersion on steel fire protection performance of epoxy-based intumescent coatings

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ABSTRACT

Epoxy-based intumescent coatings are widely used in oil and gas industries, shopping complexes and petrochemical plants to provide fire protection to the metallic substrates during the event of a fire. The present work shows how the incorporation of basalt fibers as filler material in an epoxy-based intumescent coating enhances its thermal insulation property. Dispersing agents (ethanol or glycidyl ether) were also added to the coating and their effects on the dispersion of the basalt fibers and the thermal performance of the coating were also discussed. Bunsen burner (ASTM E119) and expansion tests were performed to study the influence of basalt fibers' dispersion on the thermal insulation property of the coating. Coatings and their chars were also analysed by Field Emission Electron Microscopy (FESEM), Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD) and X-Ray Photoelectron Spectroscopy (XPS) analyses. Thermal stability was investigated using Thermogravimetric Analysis (TGA) in the pyrolysis conditions. Fire test results showed that the formulation containing ethanol as dispersing agent provided a higher fire protection, whereby the backside of the steel plate can reach a very stable plateau at 189 °C after 15 min exposure. It is also shown that the coating reached the highest expansion of 1087% with a very homogeneous char structure. The FESEM images also confirmed that basalt fibers were well dispersed when ethanol was used, whereas aggregates were formed when no dispersing agent was added. XRD and FTIR showed that the presence of boron oxide, boron phosphate, carbon and silica in the formulations which are thermally stable can improve the thermal performance of the intumescent coating. Finally, TGA confirmed that the thermal stability of formulations containing dispersing agents has been improved.

1. Introduction

In today's world, steel is the most popular and vital metal alloy being used in an offshore structure, shopping complexes and petrochemical industries. When exposed to cellulosic (ISO834) or hydrocarbon fire (UL1709) scenarios, the temperature of the steel rises sharply to an extent where its load bearing capability is decreased; above 500 °C [1], the structure collapse may occur with loss of human lifes and assets [2–5].

Various fire protection systems are available to save human lives, increase the durability of structure and to protect the assets. Intumescent coatings are designed to prevent the underlying steel substrate to reach the failure temperature. This ultimately can increase the steel integrity for 1–3 h [6]. These coatings are widely used as the passive fire protection in construction buildings, whereas maintaining the aesthetically pleasing appearance.

Intumescent coatings, when exposed to radiant heat, melt and form viscous liquid. These coatings usually contain three main active ingredients linked together by a binder. These active ingredients consist in (i) a carbonific agent, which is normally a char forming polymer or the binder itself, (ii) an acid source (ammonium polyphosphate (APP), ammonium sulphate or borates) and (iii) a blowing agent (melamine or urea formaldehyde) that can release non-combustible gases (CO_2, H_2O and NH_3) when degraded. This allows the char to swell at a certain temperature, when the system has reached the adequate viscosity. Meanwhile, some inert gases are released, from the reaction and degradation of the coating ingredients. These inert gases are entrapped in the viscous liquid thereby, form a multicellular carbonaceous structure, limiting the heat transfers between the flame and the underlying substrate [7]. The right combination of these active ingredients gives rise to the intumescent char in case of a fire [8,9]. The consistency, char thickness, thermal conductivity, mechanical resistance of the char and

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char structure are the main parameters in determining the efficiency of the char [10].

Mechanical resistance and adhesion property of the char are the main issues [11]. A wide range of reinforcing materials such as graphite fabric [12], wollastonite [10], titanium oxide [13], carbon fibers [14] and wool fibers [15] have been tested to strengthen the intumescent char [16]. For large-scale usage, reinforcing materials like fiber glass fabrics and wire meshes are also inserted in between the two coating layers. This ultimately increases the adhesion and char integrity when exposed to the fire [17]. These types of materials show some disadvantages such as require thick layers of coating, which is not cost-effective and involve high curing time.

Besides that, basalt fiber is one of the novel type of filler material which is inorganic in nature produced from basalt rock at 1400 °C. The diameter of a basalt fiber is in the range of 9–13 μm [18]. It has good thermal, chemical and corrosion resistance. The elastic modulus of basalt fiber is also high [19]. Basalt fibers are used in various applications such as automotive and aerospace industries for fireproofing textile and fabrication of pressure vessels in the chemical industries. [20]. The effect of basalt fibers on the thermal performance of intumescent coating has been conducted [21]. However, the dispersion of basalt fibers is a major issue which needs to be addressed. Basalt fibers, when used in the coating tend to cause agglomeration, which makes the coating inhomogeneous and thus affects the fire resistance property. However, incorporation of dispersing agents can enhance the dispersion of basalt fibers in the intumescent coating.

The objective of this research was to study the influence of two dispersing agents in an epoxy-based intumescent coating containing basalt fibers as the reinforcer. Fire resistance of coatings with and without basalt fibers was compared and the influence of the dispersing agents was highlighted. FESEM was used to know the char morphology, TGA was conducted to determine the thermal stability of the coatings and XPS was conducted to know the char composition in a more quantitative manner.

2. Materials and methods

Epoxy resin (NPEL-128) was chosen as the binder and polyamide amine was chosen as the hardener (H-2310, WWRC Malaysia Sdn Bhd). Ammonium polyphosphate (APP) (Exolit AP422) (Clariant Malaysia Sdn Bhd) was chosen as an acid source and swelling agent, Expandable graphite (EG) (Mc-Growth Chemical Malaysia Sdn Bhd) was used for charring and expansion agent, boric acid (Merck Malaysia Sdn Bhd) was used as an acid source and melamine (Sigma-Aldrich Malaysia Sdn Bhd) as the blowing agent. The basalt chopped strands were imported from JN Technologies Pvt Ltd. Basalt strands are mainly composed of SiO_2, Al_2O_3, CaO, FeO, MgO, Na_2O, Fe_2O_3, K_2O, TiO_2 and P_2O. The average diameter of basalt fiber was 14 μm with a length of 6 mm. The structures of basalt strand are displayed in Fig. 1. Glycidyl ether (HJ EPIOL-ME 101) and commercial grade Ethanol were purchased from WWRC Malaysia Sdn Bhd. The mild steel plates S355 ($100 \times 100 \times 1.5$ mm^3 and $50 \times 50 \times 1.5$ mm^3) were supplied from TSA Industries. They were sand blasted to remove the dust and impurities from the surface of the steel plates and to produce surface roughness that favours adhesion of the coating onto the steel.

2.1. Pre-treatment of basalt fiber

In order to remove the impurities from the surface of basalt fibers, basalt fibers were soaked in acetone for 2 h, washed with distilled water and dried in an oven for 2 h at 85 °C [22].

2.2. Preparation of coating formulations

All coating ingredients used in the formulations were weighed using AB204-S analytical balance (Mettler Toledo) according to their weight percentages. Table 1 describes four coating formulations which were studied in this research. Control formulation (CF) is the coating prepared without adding basalt fiber nor dispersing agent. Basalt fibers were then incorporated in the intumescent fire-retardant coating formulation, without adding any dispersing agent and it is coded as IFRC-R. Finally, dispersing agents (glycidyl ether and commercial ethanol) were incorporated in the IFRC-R. The amount of dispersing agents incorporated corresponds to 10 wt.% of the epoxy resin amount in the coating.

The IFRCs were prepared by incorporating the basalt fibers and the dispersing agent in the epoxy resin. This formulation was mixed using a shear mixer (Caframo) at 40 rpm for 10 min. Then, the remaining ingredients were added and properly mixed at an ambient temperature for 20 min. The prepared formulations were applied to the steel plates using the hand roller to a thickness of 1.7 ± 0.1 mm and cured at room temperature for one week.

3. Characterization of IFRC formulations

3.1. Bunsen burner test

This test was performed to study the impact of a flame onto the coating steel substrate. The test was conducted according to ASTM E119 standards [7]. A Bunsen burner fed with butane (130 g/h flow rate) was applied to the coating as shown in Fig. 2. The flame temperature was estimated using a K-type thermocouple between 100 and 1250 °C. The distance of the nozzle from the steel plate was kept at 7 cm [23,24]. The temperature at the backside of the steel plate was controlled and analysed by three K-type thermocouples stuck onto the steel plate and connected through channels to the data logger model Anarittsu Data logger, Input Channel 6 Model AM–8000 K series. The backside temperature of the steel plate was recorded for 60 min with time intervals of 1 min. For precise measurement of the data, the fire test was repeated 3 times for each sample.

3.2. Char expansion test in a furnace

In order to study the physical characteristics of the coating and char (expansion and cell structure), the samples ($50 \times 50 \times 1.5$ mm^3) were burnt in a Carbolite electric furnace (CWF 13/13) with a capacity of 13 litres ranging from 30–1300 °C. Samples were heated at a heating rate of 20 °C/min to reach a temperature of 800 °C. The temperature was held 1 h to confirm the complete burning of the samples and to reach the full char expansion, followed by cooling at room temperature inside the furnace as reported earlier [7,8]. The char expansion was measured after the furnace test.

3.3. Field emission scanning electron microscopy (FESEM)

Coatings before and after burning were examined by FESEM SUPRA 55 V P (Carl Zeiss, equipped with INCAx-act X-ray Detector, Oxford Instruments) (EHT at 5 kV for microstructural analysis; 20 kV for Electron dispersive spectroscopy-EDS). Energy dispersive X-ray spectroscopy (EDS) analysis was also performed to study the elemental composition of the char quantitatively. The cross-section view, the coating samples were cured and then cut with a sharp cutter according to the FESEM requirement for morphological analysis.

3.4. X-ray diffraction (XRD)

The residual char of the intumescent coating was analyzed by XRD performed on a Diffractometer Bruker AXS D8 Advance using Cu Kα radiation and a nickel filter ($k = 0.150595$ nm) in this range ($10 < 2\theta < 90$).