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Influences of nano bio-filler on the fire-resistive and mechanical properties of water-based intumescent coatings

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ABSTRACT

In the modern design of commercial buildings, the requirements for fire safety and evacuation must comply with to ensure the protection of human lives and property. The applications of flame-retardant materials in buildings play a vital role in reducing the risks of fire propagation. This study aims to synthesize an eco-friendly intumescent coating by incorporating the novel eggshell (ES) nano bio-filler. The samples were then characterized using Bunsen burner, thermogravimetric analysis (TGA), scanning electron microscope (SEM), Fourier transform infrared (FTIR) and pull-off adhesion tester. The coating D with an appropriate combination of the binder and flame-retardant ingredients had significantly improved the formation of char thickness in protecting the coated steel. This char layer showed a denser and more uniform foam structure surface in the SEM micrograph. Additionally, this formulation had exhibited the highest adhesion strength of 2.13 MPa, which indicated the effectiveness of interface attachment on the substrate. Moreover, the thermal stability of the formulation had also increased in thermal analysis. Therefore, the outcomes of the research revealed that uses of optimal quantity of nano bio-filler leading to better fire protective performance and mechanical properties of the intumescent coating.

1. Introduction

Fires can be catastrophic and terrifying resulting in large financial loss and human lives. The shocking deaths of at least 80 people inside Grenfell Tower due to the recent fire tragedy occurred in London, United Kingdom. The applications of fire protecting materials play a critical role to prolong the fire induced progressive collapse of building structures. Choosing the right non-combustible materials then becomes vitally important in ensuring the built environment is designed to protect its occupants in the event of a fire. Intumescent fire protective coatings offer a wide variety of versatile, durable, attractive, low thickness and low weight material in today's building structures [1]. Tremendous researches have been carried out to find a suitable solution to lessen the fire incidents in building by consideration the design of built structures and assessing its fire safety [2–6].

Intumescent coatings are insulating classification, which is designed to protect and maintain the integrity of protected substrates during a fire. This coating looks similar to ordinary paint at ambient temperature that chemically reacts to protect a building's structural steelwork, wood and concrete for a specified period in a fire. The three main eco-friendly flame-retardant additives (ammonium polyphosphate (APP), pentaerythritol (PER) and melamine (MEL) are associated together with flame-retardant fillers and binder to form intumescent coating, which acts as a thermal barrier that effectively protects the structural elements by reducing the heating rate effectively, thereby preventing the structural collapse under severe fire conditions [7,8].

The polymer binder acts as a "vehicle" film-forming element, which possess a good expansion effect and the char structure could control the migration of flame-retardant additives [9]. In this study, the waterborne vinyl acetate copolymer emulsion was chosen as an ideal binder to synthesize the intumescent coating due to its good physical and chemical properties to be mixed with other flame-retardant ingredients. The characterizations of char formation, thermal stability and adhesion strength of intumescent coatings are the main measurement techniques

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to evaluate the performance of the coating sample. Many researchers have extensively studied the performance of intumescent coatings in terms of fire resistance, mechanical properties and weather resistance [10–15].

This research highlights a renewable nano bio-filler derived from chicken eggshell waste and its huge potential in the coating industry [16]. Incorporation of inorganic and organic flame-retardant fillers play a big role in modifying the final characteristics of intumescent coatings, including its resistance to ignition, and the extent and nature of smoke and toxic gas emission products. The most commonly used nano fillers in industrial coatings are calcium carbonate ($CaCO_3$) nano powder [17]. It holds the largest market volume due to its high surface area to volume ratio, non-toxicity, good thermal stability, cost effective and easy available for industrial purposes [18,19]. Eggshell contains about 95% $CaCO_3$ in the form of calcite and 5% organic materials, which can be replaced the commercial $CaCO_3$ without compromising on the quality and effectiveness of the coatings [20–27]. Additionally, nano $CaCO_3$ is able to improve the thixotropic properties, speed up drying time, and impart gloss to the coatings. Nano $CaCO_3$ is a ubiquitous element that a wide range of applications that are of benefit from the manufacturer down to the end user.

The commercial flame-retardant fillers, such as aluminium hydroxide ($Al(OH)_3$) and magnesium hydroxide ($Mg(OH)_2$) presented a good insulating materials [28–30]. They have the ability to further decrease the heat release rate due to their low flame retarding efficiency. However, the performance of the intumescent coating system is mainly depending on the appropriate mixture of flame-retardant materials [31].

In this research work, the fire protection, thermal, and mechanical properties of intumescent coating formulations have been evaluated by identifying the appropriate combinations of flame-retardant ingredients.

2. Experimental work

2.1. Materials and samples preparation

In this experimental work, vinyl acetate copolymer with a viscosity (Brookfield RVT 3/10) of 4000 centipoises was used as a binder. This vinyl acetate acrylic binder has a good thermal stability and pigment binding properties. It was supplied by Alza Sdn Bhd in the appearance of milky white emulsion with 55 wt% solids content and particle size (median) of 0.3 μm was utilized. Ammonium polyphosphate phase II (n > 1000) (APP), melamine (MEL), and pentaerythritol (PER) were supplied by International Chemical Ltd, China. Titanium dioxide (TiO_2), aluminium hydroxide $Al(OH)_3$ and magnesium hydroxide ($Mg(OH)_2$) were supplied by Scientific Group Sdn. Bhd., Malaysia.

Different techniques and methods to synthesize the nano particles have been extensively studied and reported by many researchers as follows: precipitation of homogeneous solutions [32], water-in-oil-in-water emulsions [33], sonochemical and mechanochemical syntheses [34], and water-in-oil (W/O) microemulsions [35]. In this research project, a new and simple technique (wet milling method) in producing the ES nano bio-filler that involves four stages has been successfully developed. It includes cleaning, drying, grinding and wet milling of ES. The processes are shown in Fig. 1.

Firstly, the waste eggshell (ES) from aviculture byproduct were collected and cleaned thoroughly with water. After this, the membranes were removed from the ES. The ES were then dried for 12 h at 85 °C in the oven to produce dry and bacteria-free eggshells [18]. The dried ES were ground to a powder form by using a lapping machine. Finally, the ES powder was milled for 10 h using a high speed lab bead mill (2500 rpm) and water to obtain nano ES bio-filler. The transmission electron microscopy (TEM) was used to analyse and measure the particle sizes of the ES nano bio-filler (mean = 30.6 nm) as shown in Fig. 2.

The composition of intumescent coating was listed in Table 1. APP, MEL, PER and vinyl acetate copolymer were mixed using high-speed disperse mixer. A gauge Elcometer model A456 was used to measure the thickness of the intumescent coating to be within the range of 1.5 ± 0.2 mm.

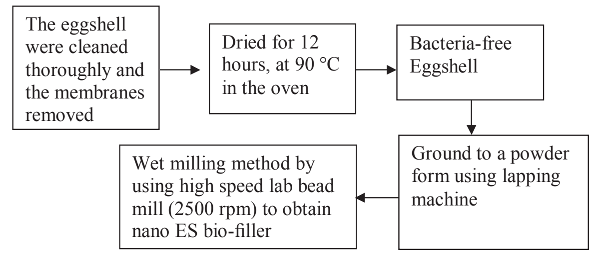

Fig. 1. Flow chart of preparing ES nano bio-filler.

2.2. Fire protection test

This test was used to characterize the formation of char thickness during and after the burning process of the intumescent coating. The fire protection test was conducted using the laboratory equipment and apparatus as shown in Fig. 3. The prepared coating was applied onto grit blasted steel plates (100 mm × 100 mm × 2.0 mm) and allowed to dry at room temperature. This process was repeated 4–6 times until a 1.5 ± 0.2 mm dry film thickness was obtained. The gas consumption of the Bunsen burner was 165 g/h. The vertically mounted steel plate coated with the intumescent formulation was exposed to high-temperature flame (about 1000 °C) for 100 min. For the Bunsen burner test, the distance between the Bunsen burner nozzle and sample was about 60 mm. Measurement of the temperature profile at the backside of steel plate 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 [2].

2.3. Scanning electron microscopy

Microscopic analyses were carried out using a tabletop Scanning Electron Microscope (SEM) (SEM; Phenom ProX desktop) to examine the surface morphology of the intumescent coatings and efficiency of char layers formation. For SEM observation, low beam accelerating voltage of 10–15 kV was operated to reduce the possibility of any thermal damage to the sample at magnification of 1000.

2.4. Thermogravimetric analysis

Thermogravimetric analysis (TGA) was carried out using a Perkin Elmer TGA 4000 model to determine the thermal degradation by calculating the residual weight of the intumescent coatings. About 5 mg of thin films were placed in a ceramic crucible and heated from 30 to 900 ºC at a ramp rate of 20 ºC per min under the airflow.

2.5. Adhesion strength

The adhesion strength test is important to ensure a strong interface bonding between the steel substrate and intumescent coating. The adhesion strength of the coated sample was determined by using the pull-off adhesion tester (PosiTest-AT-A Automatic, DeFelsko). The intumescent paint was sprayed on one side of a steel plate (dimension: 50 × 50 × 2.6 mm) with a film thickness of 1.5 ± 0.05 mm. The flat face of a pull stub (diameter: 14 mm dolly) was adhered to the coating using epoxy glue (thickness of 0.5 ± 0.05 mm). The coating surface area was measured and calculated in accordance with ASTM D4541 standard classification. The adhesion strength (f_b) for each coating specimen in MPa was calculated using Eq. (1):