



## Research paper

## Effects of talc on fire retarding, thermal degradation and water resistance of intumescent coating



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## ABSTRACT

This study presents the influences of talc as an additive in intumescent coating formulations on the thermal insulation, degradation and water resistance. The fire test was performed according to ASTM-E119 standard to study the heat shielding properties of the coated substrates. The results showed that the 20% of the talc enhanced the heat shielding and recorded substrate temperature 75 °C after 100 min of fire test. The morphology of the char was analyzed by Scanning Electron Microscopy (SEM). X-ray fluorescence (XRF), X-ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) results showed the presence of P<sub>2</sub>O<sub>5</sub>, MgO, TiO<sub>2</sub>, and SiO<sub>2</sub> respectively. The functional groups analysis of char confirmed the presence of high temperature compounds and enhanced thermal performance of coatings. Thermogravimetric analysis (TGA) showed that addition of 20% talc increased residual mass of the char by 55.55%. The lap shear test result showed that the talc also improved the adhesion of the intumescent coating with steel substrate and the highest shear strength observed was 7 MPa for a formulation, F5 containing 20% talc. Water immersion test was performed according to ASTM D870-15 and the results showed the higher water-resistance was recorded for a formulation containing 10% talc in the control formulation.

## 1. Introduction

The protection of metallic materials against fire has become an important issue in the construction industry (Weil, 2011; Gardelle et al., 2013). Indeed, the prevention of structural collapse of the building is a challenge to ensure the safe evacuation of people from the building and is a prime requirement of building regulations (Purkiss and Li, 2013) in many countries. Today, intumescent coating has been implemented widely in structure as well as highly sealed steel structure for fire protection. A passive fire retardant intumescent coating can attach to the material for a longer period and is activated when the fire or heat exists. The intumescent coating delays the heat transfer to the structure by forming an expanded protective layer or char (Ullah et al., 2013; Ullah et al., 2017b). Intumescent coatings are formulated including several active compounds which react as temperature increases and release gases (Bugajny et al., 1999). These gases cause the coating to bubble, foam and ultimately expand many times its original volume as a solid charred material. This charred material acts as an insulating barrier and protects the substrate from the rapid increase in temperature thus maintaining the integrity of the substrate structure. The

presence of charred layers slows down heat and mass transfer between the gas and condensed phases (Ullah et al., 2014a).

Intumescent polymeric systems decompose with an increase in temperature and form thermally stable carbonaceous residue (Duquesne et al., 2004; Chou et al., 2009; Ahmad et al., 2012; Ullah and Ahmad, 2014). Characteristics and performance of residue char depend on the presence of reactive additives in the coating which decompose and react with an increase in temperature. The kinetics of these reactions and synergy effects of the reactive additives and the fire performance of the coating (Wladyka-Przybylak and Kozłowski, 1999).

Clay can be used in the intumescent coating as fire retardant additive (Chuang et al., 2011; Laufer et al., 2012; Kandola and Luangtriratana, 2014; Ullah et al., 2014a). The use of the Talc has been reported in thermoplastic, Almeras et al., 2003 found that talc developed a protective layer on the polypropylene/ammonium polyphosphate/polyamide-6 composite substrate. Clerc et al., 2005 incorporated talc in magnesium hydroxide/ethylene-vinyl acetate (MH/EVA) blends to improve the fire resistant properties of these blends.

However, the effect of talc towards the thermal insulation and degradation of the intumescent coating consisting of Ammonium

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polyphosphate-pentaerythritol-melamine-titanium dioxide (APP-PER-MEL-TiO<sub>2</sub>) against steel substrate had not been studied yet. Therefore, the fire performance of this coating can be improved with Talc additive. In this study, talc had been used as an additive due to its high-temperature decomposition products such as clinoenstatite and cristobalite (Gökçe et al., 2007). This product can withstand higher temperature and its plate-like microstructure results in the cellular structure of char foam (Bakar et al., 2007; Kodal et al., 2015). Talc is a hydrated magnesium silicate with the chemical formula Mg<sub>3</sub>Si<sub>4</sub>O<sub>10</sub>(OH)<sub>2</sub> (Chin et al., 2015). Its elementary sheet is composed of a sheet of magnesium-oxygen/hydroxyl octahedral, sandwiched between two sheets of silicon-oxygen tetrahedral.

The objectives of this work are to investigate the effects of talc on the thermal insulation and the thermal degradation and water resistance of the intumescent coating (APP-PER-MEL-TiO<sub>2</sub>). The performance of talc had been investigated on heat shielding, char expansion, morphology, char residue composition, thermal degradation of formulations, the elemental composition of char and functional groups. Furthermore, the effect of talc on the adhesion of the coating with the substrate and the results of char expansion, and char morphology after the water immersion test were also discussed.

## 2. Materials and method

Epoxy resin Bisphenol A was purchased from Chang Chun Plastics Co. Ltd., ACR hardener H-2310 was purchased from ACR Tech Co Ltd., Zinc Phosphate Primer (Commercial Grade), Ammonium Polyphosphate (APP) purchased from Clariant (Exolit AP 422), Pentaerythritol (PER) purchased from Merck, Melamine purchased from Sabic Chemical Industries, boric acid, TiO<sub>2</sub>, talc with particle size 30 mesh were purchased from Sigma-Aldrich. The SEM image of talc is presented in Fig. 1.

### 2.1. Preparation of the coating substrate

The ingredients of intumescent coating are listed in Table 1. A freshly grounded mixture of all the ingredients was mixed with epoxy and hardener. The coating mixture was stirred at 40 rpm for 30 min for the homogeneous mixture. Steel substrate was sandblasted and the surface roughness was determined by profilometer and the surface roughness average was Ra 2.5. The dimensions of steel cross-section 10 × 10 cm<sup>2</sup> were used. Epoxy zinc phosphate primer was used as interlayer in this work to provide corrosion protection and improve the adhesion of the coating with steel substrate. The intumescent coating was applied using a brush on the steel substrate and an average thickness of the coating was maintained at 1.5 mm and it was measured

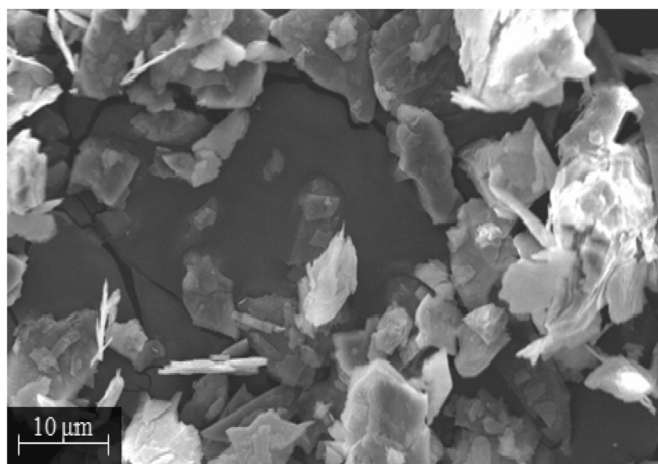


Fig. 1. Microstructure of the talc used in this work.

**Table 1**  
Composition of intumescent coating (mass%).

Coating formulation	F1	F2	F3	F4	F5
Epoxy	25	25	20	20	15
Hardener	25	20	20	15	15
APP	18	18	18	18	18
PER	6	6	6	6	6
MEL	6	6	6	6	6
Boric acid	15	15	15	15	15
TiO <sub>2</sub>	5	5	5	5	5
Talc	0	5	10	15	20

by digital vernier caliper. The coated substrates were cured in the oven at 70 °C for overnight.

## 3. Characterization techniques

### 3.1. Furnace fire test

To analyze the char expansion and morphology after fire test, the intumescent coating samples were burnt in a carbolite furnace, model no CWF 13/13 have a temperature range 30–1300 °C with chamber capacity 13 l. The furnace was operated under air at a heating rate of 20 °C/min to achieve 800 °C and this temperature was maintained for 60 min to ensure the complete burning of the samples. The char samples were cooled in the furnace and char expansion was measured after furnace fire test and compared with coating thickness. The char expansion was measured using digital vernier caliper at four different location of the char and the average char expansion is taken with considering error ± 0.05.

### 3.2. Heat insulation test

Fire test was conducted to evaluate the penetration of fire to the steel substrate according to ASTM-E119 (ASTM and ASTM E119-16a, 2016) and the test was carried out for 100 min. Butane gas burner was used to burn the coating and the distance of the burner from the coating substrate was set at 7 cm. The applied temperature of butane Bunsen burner was 950 °C. K-type thermocouples were connected to Anaritsu Data logger, Input Channel 6 Model AM-8000K with Anaritsu software and were placed on the back side of the steel plate with dimension 10cmx10cm. The temperature of the backside steel plate was measured at an interval of 1 min.

### 3.3. Field emission scanning electron microscopy (FESEM)

Char samples prepared by burning of coating at 800 °C for 60 min in a carbolite furnace were examined for morphology under FESEM model SUPRA 55VP manufactured by Carl Zeiss AG, Germany. The acceleration voltage was 0.1–30 kV and working voltage was 4–5 kV. The char morphology was observed at 500 × magnification.

### 3.4. X-ray fluorescence (XRF)

The elemental composition of the coatings and residual char was studied by XRF. All XRF measurements were carried out with a wavelength dispersive XRF S4 Pioneer (Bruker AXS, Karlsruhe, Germany). The S4 was equipped with Rh tube, different copper foils (100 and 200 μm) as primary filters, five crystals with different ranges and resolutions and two different detectors (flow counter and scintillations counter). The powder sample of the coating and the char was compacted in a specific container using a compactor before spectrometer measurements.

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