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Preparation and performance of waterborne intumescent coatings with mechanical, thermal properties and optimization of flame retardant paints

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Many industries typically like chemical industry, petrochemical and oil refineries etc. are prone to fire. In such a case intumescent (IMT) paint will provide extra protection to substrate wherever it is applied. The aim of present work was to optimize the fire protection performance and thermal properties of water-based IMT fire protective coatings on steel structures through the use of flame-retardant paints. The IMT coating paint was prepared using water-based styrene acrylic emulsion (SAE), flame-retardant additives (Ammonium phosphate, Pentaerythritol, Melamine and Dicyanadamide and flame-retardant fillers (TiO_2 and $CaCO_3$). All these ingredients are mixed using mortal pestle. The coating paints with and without fire retardant have been characterized by studying chemical properties by using FTIR, mechanical properties (Flexibility test, Impact resistance test and Pull off test) and thermal properties (Bunsen burner test, furnace test, and Thermogravimetric analysis test). The results demonstrated that mild steel specimens coated with IMT paint exhibited effective fire protection performance; excellent adhesion strength, improved uniform char layer formation, and enhanced thermal stability. It revealed that fire retardant coating had better or same properties than without fire retardant coating. Hence, formulated IMT coating paint proved effective in protecting steel structures against fire.

Keywords: Adhesion strength, Flame-retardant additives, Flame-retardant fillers, Intumescent coatings paint, Styrene acrylic emulsion, Water resistance

Introduction

One of the most threatening hazards to the safety of people and property has always been fire[1,2]. At high temperatures of fire, it disrupted the structure of materials due to local instability and accelerating flames[3]. Therefore, it is important to treat flammable materials with flame retardant[4,5]. Water-based fireproof coatings can swell due to the heat-induced interaction of their micro-components, which results in thinner coatings, quicker curing and moulding, better fire resistance, etc[6]. To ensure fire safety and construction laws in many countries, intumescent (IMT) fire protective coatings have been widely employed as passive fire protection in steel structures, building components, automobile parts, and airplanes[7]. One of the most effective ways to protect various substrates from fires is to apply IMT coatings. Because of its many advantageous qualities, like being lightweight, odourless, and environmentally friendly, IMT paint is newest trend in fire retardant building materials[8,9]. In case of a fire, steel tends to bend and loses its structural qualities at 500°C, which can cause building structures to collapse[10]. The application of passive fire protection materials can significantly extend fire resistance time by insulating steel structures from elevated temperatures during fire exposure[11]. The fire resistance period can be extended by using passive fire protection materials that insulate steel structures from the effects of high temperatures generated during a fire. Although many linger widely on steel structure applications, extensive research has shown that the IMT flame-retardant coating possesses favourable flammability characteristics along with strong physical and chemical properties[12,13].

The IMT flame retardant is a multi-phase green flame retardant system with two aspects to its flame retardant mechanism[14]. Firstly, condensed phase flame retardant in which carbon porous layer created by burning can shielded the energy transfer and raised the fire resistance limit of the matrix[15]; secondly, gas phase flame retardant, which lowers the concentration of flammable gas by producing inert gas during combustion[16]. Although water-based IMT paint can greatly improve the material's fire suppression performance. Defects like excessive heat generation during combustion, a high smoke production rate and low coke strength of IMT fire retardant coating itself have limited application in flame retardant fields[17]. The IMT coatings are generally classified into two types such as solvent-based and water-based IMT coatings. The experimental study primarily focuses on evaluating the thermal properties and fire protection performance of water-based IMT coatings. The coating is a user-friendly alternative to solvent-based options due to its low toxicity and greater environmental friendliness. During a fire, when the temperature rises between 280 and 350°C, intumescence forms as the coating degrades in the melt zone[18]. As the temperature further rises between 350 and 420°C, the IMT coating undergoes decomposition in the reaction zone. At temperatures above 420°C, a carbonaceous porous char layer develops, providing excellent heat insulation that protects the underlying steel for 1 to 3 h (charring zone)[19]. This saves more lives by giving enough time for evacuation. Optimizing the formulation is essential to create an efficient char layer with high durability and a uniform foam structure to effectively protect the substrates[20]. Present research work has also highlighted huge potential for integrating the IMT coating components, which include styrene acrylic emulsion (SAE) is a versatile polymer often used in coatings, paints, adhesives, and other applications where a durable, flexible, and water-resistant film is required. Ammonium phosphate [$(NH_4)_3PO_4$], an acid source; pentaerythritol ($C_5H_{12}O_4$), a carbon source; melamine ($C_3H_6N_6$), a blowing agent; and dicyanadamide ($C_2H_4N_4$), a curing agent, are the four main flame-retardant additives. These flame-retardant additives are mixed with titanium dioxide (TiO_2) and calcium carbonates ($CaCO_3$) are flame retardant fillers; and SAE combine to provide a protective thermal barrier at high temperature on steel structure. The binder plays a crucial role in the IMT preparation process since it promotes the expansion of the char layer and ensures the formation of uniform char foam structure[21,22].

Several benefits of applying IMT coating paint over conventional structural fire protection techniques, giving steel work an attractive surface, quick application, simplicity in handling intricate details, and maintaining the intrinsic properties of steel structures[23]. This study primarily investigates how varying compositions of flame-retardant additives affect the fire protection performance and thermal properties of water-based IMT coatings. Over a few decades, in the field of fire protective coatings commonly use IMT flame-retardants because they showed exceptional flame-retarding performance when applied on a variety of materials, including steel, wood, wires, and even polymers. The flame-retardant chemicals play a significant role in producing the light char layer. Such char layer possesses a very low heat conductivity to prevent the fire from spreading to substrate. In addition, fillers are needed to strengthen the char layer structure coating by including resistance of fire ignition, slow down the rate of char layer growth, and minimize gas emissions. This is because the char layer that forms easily oxidized and has poor adhesion strength[24]. Therefore, fire-retardant fillers are incorporated to improve several critical performance aspects of IMT coatings, such as thermal stability, char strength and compactness, flame-retardant efficiency, smoke suppression, mechanical integrity of the expanded char layer and adhesion to the substrate.

There are numbers of fire retardant fillers which are widely used such as magnesium hydroxide, calcium carbonate, titanium dioxide and aluminium hydroxide[25]. Binders play a crucial role in supporting the formed char and preventing its collapse. It also facilitates softening and char formation during a fire. This work investigates the effects of flame retardant additive composition on the mechanical characteristics and fire safety performance of a water-based IMT coating during an event of with fire and without fire. The coatings paint with and without fire retardant will be investigated through several experiments such as flexibility test, impact resistance test, pull off test, Bunsen burner test, furnace test, and thermo gravimetric analysis test and flourier transform infrared analysis.

Experimental Section

Preparation of intumescent coatings paint

Water-based IMT coating paint was formulated using a mortar pestle mixed all components for 30 min at room temperature until it is completely homogenous. A laboratory-scale mortar and pestle batch consisted of approximately 100 g of total

Table 1 — The compositions of coatings paint weight in % (A) without fire retardant and (B) with fire retardant additives

Ingredients	Chemicals	(A)	(B)
Water-based polymer binder:	SAE	80	40
Fire-retardant additives:	APH	--	20
	MEL	--	15
	PET	--	10
	DCM	--	05
Fire-retardant fillers:	TiO_2	12	06
	$CaCO_3$	08	04

material. The components were gradually added and manually ground for 10–15 min to ensure uniform dispersion of the solid additives. Homogeneity of the mixture was verified by visual inspection for uniform colour and texture, absence of agglomerates, and consistency across repeated batches. The reproducibility of the coating quality was confirmed through repeated mechanical and thermal tests, which showed negligible variation among batches[26].

The composition of IMT coatings are shown in Table 1. The IMT formulation was made up of 100 wt.% of ingredient. Polymer binder 40 wt.%, flame-retardant additives 50 wt.% and fillers consist 10 wt.%,. The chemicals ammonium phosphate (APH), pentaerythritol (PET), melamine (MEL) and dicyanadamin (DCM) were used as flame retardant additives. The TiO_2 and $CaCO_3$ were used as flame retardant fillers, and the SAE serves as a water-based polymer binder. The SAE was supplied by Jesons Industries Ltd. India. The APH, PET, MEL and DCM were purchased from Merck, India. The TiO_2 and $CaCO_3$ were purchased from Sigma-Aldrich. The fire resistance and physical properties of the IMT coatings were characterized and assessed through a number of tests, including the flexibility, impact resistance; pull off, FTIR, Bunsen burner, furnace, and thermo gravimetric analysis test.

FTIR analysis

Fourier transform infrared (FTIR) spectroscopy is typically used to identify the chemical structures and functional groups that are present in a test sample. FTIR spectra were used to determine the functionalization of the prepared IMT coating sample in 40% water-based polymer in comparison to without fire retardant coating.

Flexibility test

In accordance with ASTM D-55293, the flexibility of the coated paint formulation was tested using a ¼" dia. conical mandrel tester. Place the panel so that one short edge hits the small end of the mandrel and the coated side faces the draw bar. Within 2 to 3 s, clamp the panel and use the draw bar to bend it 180° evenly and without jerking over the mandrel[27].

Impact resistance test

Using an impact tester (ERICHSEN GMBH & CO, D-5870, Model304), impact resistance of the coatings was assessed in accordance with ASTMD2794. As is standard, the coated mild steel specimen was dropped from a height of 65 cm while being subjected to an impact weight of 0.908 kg. The scratch resistance test of coatings was performed in accordance with BS-3900 standard, and the test area was inspected for cracking and coating loss of adhesion[27].

Pull off test

The pull-off method was used to assess adhesive strength of coating paint sample in accordance with Indian Standard IS: 101. In order to make it easier to de-bond the coating from one side, the technique was modified by reducing the area of one dolly. A Universal Testing Machine (Lloyd, Model-LR30K) was used for the measurements[27].

Bunsen Burner Test

On a steel plate measuring 150 mm (L) × 75.5 mm (W) × 1.5 mm (H) and having a thickness of 800 ±50 microns, the IMT coating samples were coated both with and without fire retardant. The backside temperature of steel plates with and without IMT fire-retardant coating paint was measured using an infrared thermometer. The sample was heated to approximately 400°C for 45 min using a Bunsen burner flame blow torch. The samples and the Bunsen burner flame are in close proximity[28].

Furnace Test

The thickness of the char coating generated at 500°C was measured and observed using a furnace test. The coating paint was coated with fire retardant and without fire retardant on 150 mm × 75.5 mm × 1.5 mm steel plate with a thickness of 800± 50 micron and left to dry for one week before conducting the furnace test. After that, the coated samples were put in a furnace that raised the temperature from room temperature to 500°C, all panel placed simultaneously in furnace for 45 min. Then, thickness of the char layer for each sample was determined.

Thermo gravimetric analysis test

The coated paint's thermal stability was determined by thermo gravimetric analysis (TGA) using thermo gravimetric-differential thermal analyzer under nitrogen gas atmosphere. In accordance with ASTM E1131, the samples were heated in aluminium pans from room temperature to 500°C at a rate of 10°C per min. All thermal degradation data was obtained from the TGA curves.

Results and Discussion

To confirm the poly-condensation reaction of SAE and flame-retardant additives utilized in this study, the coated paint formulation with and without fire retardant is discussed.

FTIR analysis

Fourier Transform Infrared Spectroscopy (FTIR) used to analyse fire retardant paint containing specific chemicals like APH [$(NH_4)_3PO_4$], PET ($C_5H_{12}O_4$), MEL ($C_3H_6N_6$), and DCM ($C_2H_4N_4$). These compounds are commonly used in fire retardant systems for their ability to form protective char layers, release non-flammable gases, and enhance thermal stability. FTIR analysis is typically to study the chemical interactions, reaction products, and overall effectiveness of these materials in improving the fire resistance of the paint.

The APH is an IMT fire retardant that releases phosphoric acid when heated, which helps to produce a char layer on the substrate, insulating it from further heat. FTIR used to identify the characteristic phosphate ($–PO_4$) and ammonium ($–NH_4$) functional groups, confirming the presence and concentration of ammonium phosphate in the paint. The PET is often used as a charring agent. When exposed to heat, PET helps the formation of a stable carbonaceous char. FTIR would help identify the hydroxyl (–OH) and ether (–C–O–C–) groups in PET and assess how these groups participate in the char formation process under thermal conditions. However, MEL is used in fire retardant systems for its ability to generate nitrogen gas when heated, which dilutes flammable gases and slows down combustion. FTIR can detect the amine ($–NH_2$) and amide (–C=O, N–H) functional groups in MEL and monitor how these groups decompose or react at elevated temperatures to form flame-retardant products.Another nitrogen-containing substance that can improve coatings' flame resistance and thermal stability is DCM.FTIR analyze the cyano (–C≡N) and guanidine (–NH–C=NH) functional groups, which are responsible for the compound's behavior when exposed to heat.The FTIR results will primarily reflect the composition of the base material (e.g., SAE), allowing identification of the material's chemical structure. Peaks corresponding to the backbone of the polymer will be prominent. The FTIR data can also help assess the stability and chemical bonding within the material. A sample without fire retardant might show standard chemical characteristics that are less influenced by thermal stability or degradation.

The FTIR spectra (Fig. 1) of fire retardant-treated materials usually show characteristic peaks that represent both the base material and the added fire retardant chemicals. In polymer-based materials, peaks of C–H stretches (3000-2855cm^{-1}) and C=O stretches 1085-1000 cm^{-1}) are shown in Fig. 1. The appearance of N≡C stretching vibration (2170-2130 cm^{-1}) suggests that incorporation of MEL into the polymer matrix. If hydroxyl group is present in fire retardants (e.g., pentaerythritol compounds) are used, new peaks related to O–H stretches (around 1400-1300 cm^{-1}) indicates hydroxyl group incorporation. Changes in polymer backbone: The intensity or shift of certain peaks can suggest the interaction between the fire retardant and the polymer, such as a possible cross linking effect, or physical changes in the polymer structure (e.g., a decrease in the intensity of the C=O stretching frequency in case of esterification). The appearance of certain functional groups, such as –OH, $–PO_4$, $–NH_2$, or –COOH, can reveal information about the flame retardant process. For example, the presence of –OH or $–PO_4$ indicates the formation of a char layer during combustion that helps lowers flammability.

The FTIR spectra could provide evidence for the development of a protective char layer that prevent heat transfer. For instance, a shift or decrease in carbonyl absorption (C=O stretching) might indicate the

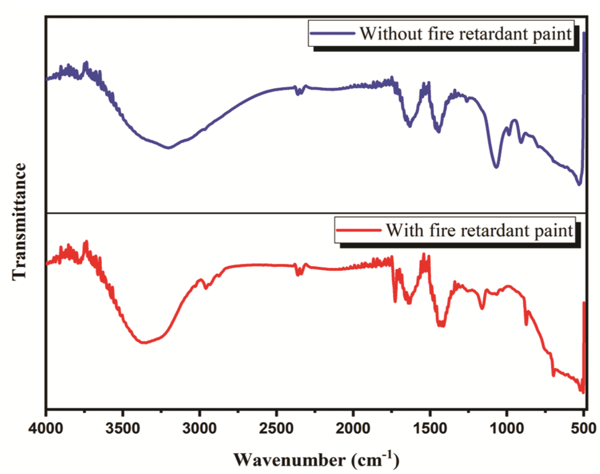

Fig. 1 — FTIR spectra of samples without and with fire retardant

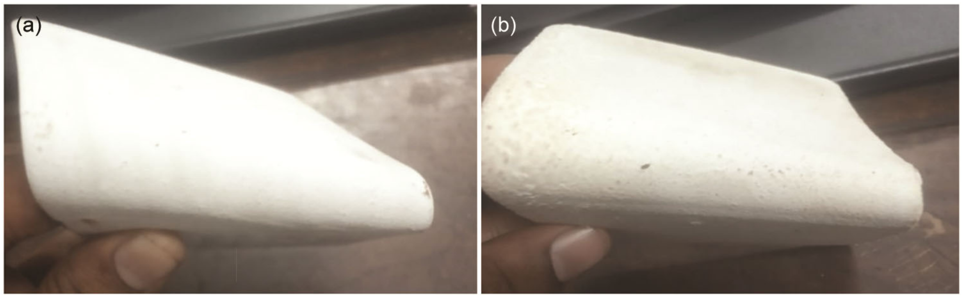

Fig. 2 — Flexibility of samples (a) without and (b) with fire retardant

formation of a charred structure that helps in fire resistance. The appearance of phosphorus or nitrogen-containing groups (e.g., NH_2, PO_4) may indicate that the fire retardant works by releasing non-flammable gases like ammonia or phosphoric acid, which helps in flame inhibition and suppression of the combustion process. The FTIR can reveal any synergistic effects between the fire retardant and the polymer. For example, multiple functional groups present in the fire retardant compound may lead to an elevated the effect, where the interaction between different chemical groups improves the fire resistance. For instance, phosphorus and nitrogen together combination could result in a formation of more stable char. FTIR can show whether improved the thermal stability of the treated material. Peaks associated with polymer backbone could show reduced intensity or new formations that indicate degradation at higher temperatures. This would suggest that the fire retardant is protecting the polymer by stabilizing it during heat exposure.

Flexibility and impact resistance analysis

Steel specimens were coated with and without fire retardant using water-based coatings, and the flexibility and impact resistance behaviours were evaluated in accordance with the requirements described in the section on "Scratch and impact resistances." According to a previous study, aqueous polymer coatings have the benefit of being more flexible and curing at room temperature[29]. Similar to this, the reactive technique preserves the natural flexibility of the water-based polymer coating by creating a waterborne polymer by grafting or adding flame-retardant components or groups into the water-based polymer molecular chain[30].The coated samples in the present study, both with and without fire retardant, passed the bent in a mandrel with diameter of ¼ inch. The existence of a flexible polyester chain containing SAE, which reduces its brittleness, is responsible for this flexibility. The results are shown in Fig. 2.

Impact testing demonstrates that the water-based polymer permits the impact of its weight and produces excellent consequences. A previous study examined the effects of mixed carbon materials, including graphene and carbon nanotubes, on the fire-retardant qualities of waterborne acrylic-resin-based IMT fire-retardant coatings. The results showed that the combination of graphene and carbon nanotubes produced a synergistic interaction that greatly enhanced fire protection and thermal stability[31]. In our study results the prepared coating sample both with and without fire retardant was applied on mild steel specimens and assessed for scratch hardness resistance properties according to the criteria outlined in the "Mechanical analysis" section. After testing with and without fire retardant coatings paint comparable hardness as shown in Fig. 3. There is no hardness was found with and without fire retardant coating sample. Hence, impact resistance test passed in both the coating sample.

The more polar structure of the polymer created by including the aliphatic ester and acetate group of SAE was found to greatly improve the adhesion of water-modified polyester coatings on metal surfaces. Previous report shows that waterborne IMT coatings have steadily surpassed solvent-based coatings in terms of adherence and endurance[32]. Moreover, waterborne epoxy resins are widely used as film-forming matrices for IMT fireretardant coatings due to their exceptional adhesion and chemical resistance[33]. In our study steel plates were coated using water-based polymers with and without fire retardant. Fig. 4 shows that coating with fire retardant paint indicated good adhesion properties compared to without fire retardant coating.

Bunsen burner test

After 60 min of fire testing, the temperature profiles of the mild steel plate and the steel plates coated with and without fire retardant coating sample were compared. The critical temperature for the coated sample, both with and without fire retardant, was determined to be 400°C[27].The fire protection outcomes of coated steel plates were plotted as a function of time and results are shown in Fig. 5.

The digital handheld thermometer was used to record the temperature profile during the fire exposure. Moreover, thickness of char layer after burning has measured in order to check the fire resistance performance of coated paint. The coated sample would ultimately reach its equilibrium temperature and maximum char layer thickness throughout the heating process. Hence, equilibrium temperature and thickness of char layer formed are used to indicate the fire protection performance of coating samples. Therefore, the char layer thickness and equilibrium temperature are used to show the fire protection performance of coating samples.

The coated samples exhibit a similar temperature pattern, falling below 300°C, as demonstrated during the first 10 minutes. Then the temperatures started to fluctuate at 20 min due to chemical reactions of the coating formulations during the high-temperature test. The temperature increase continues for 45 min, it has been observed that the coating with fire retardant to enhanced rapidly until reaching temperature 450°C at 47 min. This might be because, during the fire test, the coated sample began to lose its interfacial bonding or adhesion strength to the steel plate. After reaching temperature 400°C at 47 min, the fire retardant coating rapidly lowers; this could be because the gas pressure from the Bunsen burner flame spray gun blow torch is reduced. Additionally, it is noted that the temperature of both coating plates begins to fluctuate again until it reaches 50 min. The temperature reaches equilibrium after 50 min of testing and almost remains unchanged until the last phase of the test. This study shows that in good agreement with other researchers, the char combined the beneficial effects of SAE, flame retardant

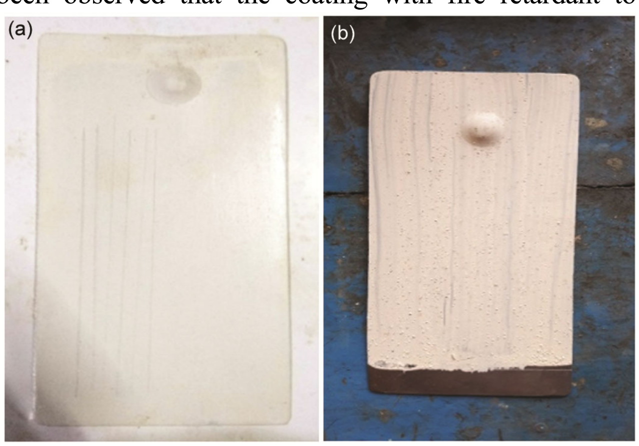

Fig. 3 — Impact analysis of samples (a) without and (b) with fire retardant

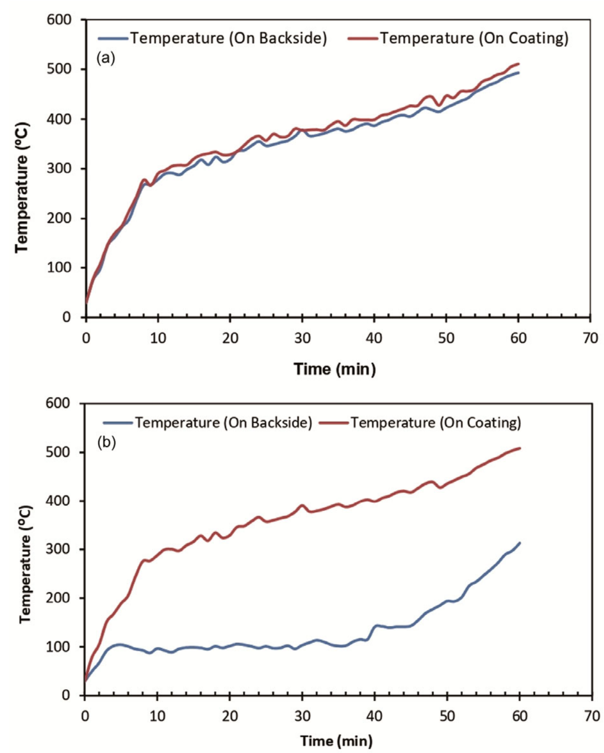

Fig. 5 — Temperature profile on the coated steel plates after the Bunsen burner test; (a) without and (b) with fire retardant (Thickness of steel plate = 1 mm and thickness of coating = 100 micron)

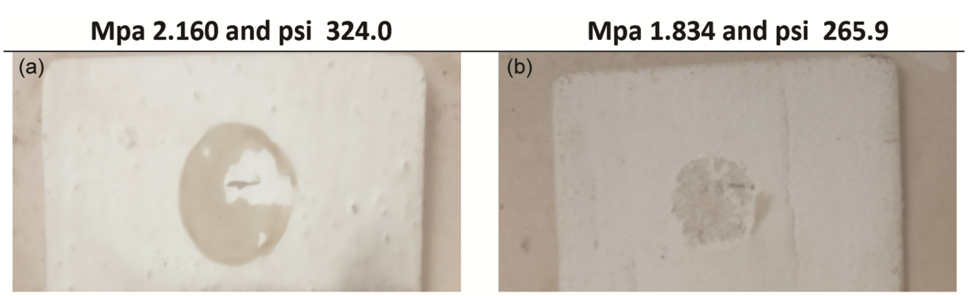

Fig. 4 — Pull off test of samples (a) without and (b) with fire retardant

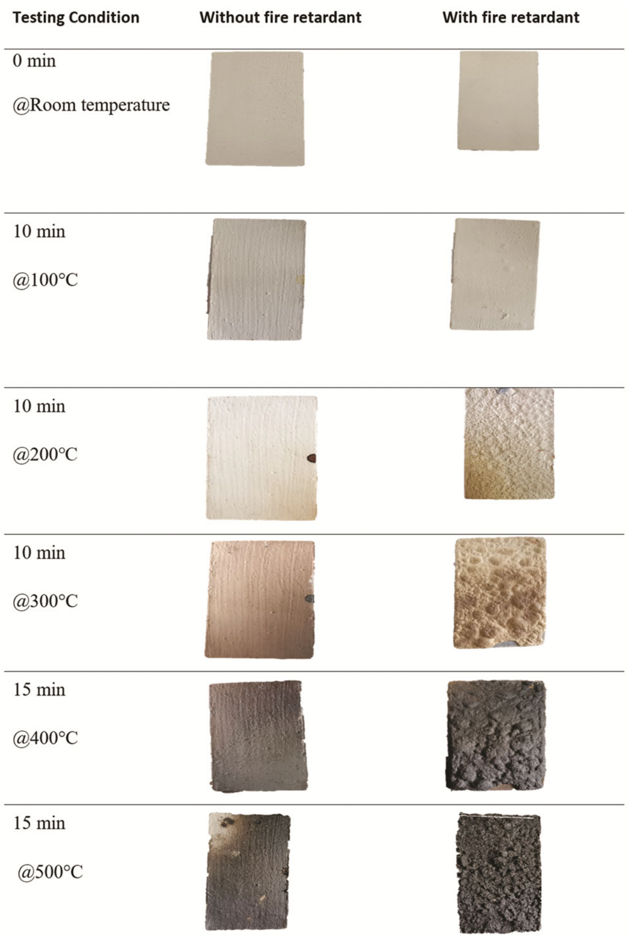

Fig. 6 — Furnace test results

and fillers to create the thickest and best char layer (approximately 6.0 mm) to prevent the formation of a uniform foam structure and promote a good fire-protective barrier.

Furnace and TGA

Coating samples with and without fire retardant additives was heated under the temperature 500°C and char layer thickness was observed and results are shown in Fig. 6. The IMT coating with fire retardant formed the thickest char layer as compared to without fire retardant coating sample which indicated that coating with fire retardant had the best char layer expansion. The IMT coating with fire retardant reached 15 mm of char thickness at 500°C. This indicates that excessive amount of APH may inhibits the expansion of char layer. Hence, it can be deduced that too much APH could restrict the char layer formation. Therefore, optimization of APH, PET, MEL and DCM concentration plays a significant role in providing the best fire protective performance.

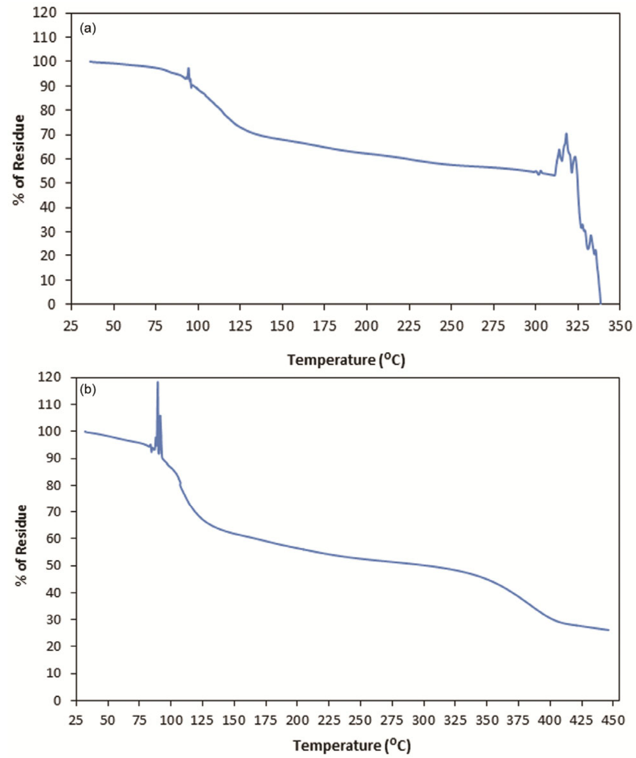

Fig. 7 — TGA curves of coating sample (a) without and (b) with fire retardant

The thermal degradation of coatings paint was determined and analysed using TGA. Previous research shows that the gas-phase flame-retardant mechanism includes preventing free radicals from causing chain reactions during burning[34]. It is possible to effectively inhibit combustion by slowing down the heat release process and partially preventing the pyrolysis of materials by stopping free radical reactions. During combustion, the flame retardants in the IMT system undergo thermal decomposition to release inert gases such as carbon dioxide, ammonia, and nitrogen[35]. Consequently, oxygen content and concentration of flammable gas in the combustion atmosphere are significantly decreased. Furthermore, by absorbing heat during the chemical breakdown process, the flame retardant lowers the reaction temperature and slows the spread of combustion, which intensifies the flame-retardant effect.In the present study coating samples with and without fire retardants were heated up to 500°C to observe the weight lost due to thermal degradation. The thermal decomposition was observed at temperature 500°C based on the data shown in Fig. 7. The graph curves observed to be slightly moved towards higher temperature ranges with moderate changes in heating rates, despite the profiles being very comparable. The graph Fig. 7(a) signifies that TGA graph for without fire retardant, which does not contain fire retardant additives, exhibits a slightly different pattern after reaching 150°C. Between 150°C to 325°C, the coating shows a loss of around 10% in mass. However, above 300°C, the decomposition of the binder (Styrene-acrylic Emulsion) leads to a sudden mass loss. The temperature range between 325°C to 450°C shows a loss of 40% mass. At a temperature of 340°C, the coating exhibits 10% mass residue due to the high loading of filler, such as $CaCO_3$ in IMT coating sample. These findings suggest that the lack of fire retardant additives in the coating paint significantly affects its ability to withstand high temperatures, with the decomposition of the binder being the main cause of mass loss.

Fig. 7(b) reveals that sequence of the chemical processes that occur in the with fire retardant, Temperature below 125°C, shows about 30% mass loss due to water evaporation. Temperature between 150 and 225°C the release of polyphosphoric acid and ammonia from the APH, temperature between 225 and 280°C, etherification of the source of carbon pentaerythritol by polyphosphoric acid and the formation of the structures of future phosphorus-carbon frame. The temperature range 280–350°C, re-grouping of polyphosphoric ethers with the further formation of a coke layer may occur. This chemical changes occur in range 150 to 300°C, in that range only 15% mass loss is observed. The blowing agent MEL decomposes in temperature 260 to 280°C into component like DCM. The temperature above 300°C, it is observed that sudden loss in weight that foam the melting coke layer and organize the porous structure of the swollen coating. However, in general, the thermal degradation temperature of SAE is typically between 200 and 300°C. This means that the emulsion can begin to break down and lose its properties at these temperatures. The graph of TGA shows that IMT is fully decomposed at about 500°C.The addition of TiO_2 and $CaCO_3$ fillers to the IMT binder may improve anti-oxidation properties of coating, as the TGA curves clearly shows that the coating sample without fire retardant has the highest residual weight.

Conclusion

The following conclusions can be deduced from the series of experimental tests. The mechanical characteristics and fire-protective performance of the IMT coating can be directly impacted by the appropriate combinations of flame-retardant materials chosen. It has been determined that the water-based IMT coatings with 20% APH, 15% MEL, 10% PET, and 5% DCM added as fire-retardant additives, coupled with 6% TiO_2 and 4% $CaCO_3$ bio filler, have the best thermal, mechanical, chemical, and fire-protective performance. Therefore, it can be revealed that an appropriate composition of flame-retardant additives effectively enhances both fire and weather resistance in the protection of steel structures.

Conflict of interest

The authors declare no conflict of interest.

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