Fabrication of Intumescent Fire-Retardant Coating for Structural Applications

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Abstract - Applicability of materials limited by the effect of flammability in case of any sudden incident. Worldwide protection of any structure from the fire is covered through intumescent fire resistant coatings since many past years. Purpose of this study is to prepare and to fabricate several intumescent fire retardant coatings (IFRC) using different proportions of materials per unit weight. Also to find most optimistic formulations and analyse the performance of intumescent fire retardant coatings (IFRC). On the basis of background studies 5 different materials were considered to prepare several formulations i-e Titanium dioxide (TiO_2), Ammonium polyphosphate (APP), Zinc borate, Epoxy hardener, Kaolinite (Clay), Melamine (MEL) and Expandable Graphite (EG). IFRC formulations were applied on Mild Steel plates after its preparation Each formulation was subjected to a temperature of 700°C for a period of five minutes. The results revealed that the coatings, Out of 5 formulations of IFRC, IFRC-4 exhibited promising performance. It demonstrated excellent stability and adhesion at the elevated temperature, lasting for over five minutes. Additionally, its coating expanded up to four times the initial thickness. Heat Transfer rate was completely reduced.

***Keywords*:** Epoxy hardener, Melamine (MEL), Titanium dioxide (TiO_2), Ammonium polyphosphate (APP), Expandable Graphite (EG), Intumescent fire retardant coatings and Kaolinite (Clay)

1. Introduction

In recent years, intumescent fire-resistant coatings have gained widespread use in various materials to meet the growing demand for improved fire safety parameters to reduce the potential fire risks in many fields, such as textiles, wood, steel, plastics, and others. The purpose of coating application can be either for decorative or protective reasons. The coating itself can cover the entire substrate or only selected portions of it. On the basis of usage of curing agent and resin type, these coating substrates are identified [1-2].

Fire retardant coatings are among the simplest, ageing, and to the highest degree effective ways to defend surface of materials against burning. This method involves creating a preventive layer that changes the heat flux to the substrate and may limit its change of state thermally, flaming, or burning. The best fire-resistant coatings have a low fire spreading, minimal or lower release of harmful toxic gases/smoke, are easy to fabricate and apply, have good ability to resist against wear, cost-effective and well correspond to the fundamental substance [3].

It's important to note that the meaning of fire-retardant reflects the difficulty in burning, it does not describe the ability to keep material safe from burning or unburnt. Self-extinguishing ability of fire-retardant substances after burning differentiate other fire-retardant substances which ignite only once by keeping the burning rate of the main object slow [4]. Intumescent coatings and non-intumescent coatings are the main category on the basis of which fire retardant coatings are classified.

Intumescent coatings have the ability to expand when exposed to high temperatures, creating a layer of burnt cells that act as a barrier to reduce the transfer of heat and mass between the solid and vapour phases. This layer provides protection to the substrate. When exposed to fire, intumescent fire-resistant coatings increase in volume by 20 to 30 times, degrading coating as the fire continues. Intumescent fire retardant coatings (IFRCs) are a type of reflexive fire protection system that not only prevents fire ignition but also slows down the spread of fire, thereby increasing evacuation time in the event of a fire. IFRCs consist of three active components, including a source of acid , a blowing agent and a substance of carbon. In fire protection systems APP, EG, and MEL are commonly used. When exposed to heat, the intumescent coating expands and decreases in density [3].

When ammonium polyphosphates are used as flame retardant in polymers, it possesses lengthy chains and a specific crystallinity known as Form II. Upon reaching a temperature of 240°C, these polyphosphates undergo decomposition and produce ammonia and oxyacid [5]. When epoxy comes to heat resistance, room temperature cured epoxy plastic and heat cured epoxy plastic exhibit minimal differences. The HDT (Heat Deflection Temperature) or Tg (Glass transition temperature) are the typical measures of heat resistance. However, the mechanical strength of the plastic decreases quickly at HDT. The HDT for room temperature cured epoxy plastic usually does not exceed 70°C, while heat cured epoxy plastic can withstand temperatures up to 250°C [6]. The production of expanded graphite begins with immersing natural flake graphite in chromic acid, which is then followed by concentrated sulfuric acid. This process causes a crystal lattice expansion, resulting in expanded graphite. The expanded graphite material has the ability to char and expand during a fire, thus preventing fire penetration and propagation [7]. Melamine possesses exceptional capability of retardancy of flame owing to its property to perform in several ways against all steps of combustion process. Endothermic dissociation is a phenomenon in which melamine acts as a heat absorber to prevent flaming. In the subsequent stage, the breakdown of melamine vapours generates an even more pronounced heat sink effect [8]. Titanium dioxide is in high demand globally, with around 80% of the demand coming from the paint and varnish industries, as well as paper and plastics. Other applications, including fibres, rubber, printing inks, cosmetics, and food, make up approximately 8% of its usage [9].

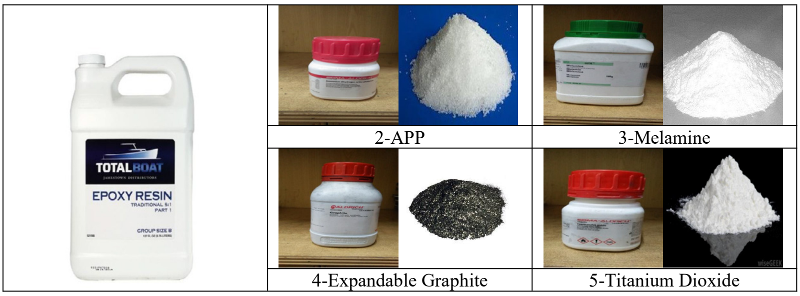
Zinc Borate is a flame retardant that contains boron and is compatible with a variety of polymeric materials. It exhibits exceptional flame retardant properties in both solid and gas phases, and its smoke-suppressing ability aids in hastening rescue time during a fire. Zinc Borate is a versatile flame retardant that helps to establish a protective glassy layer and sturdy char layer, reducing the production of dangerous smoke during a fire [10]. Kien-Sin Lim et al. [11] have shown the usage of APP (ammonium polyphosphate) in several kinds of composites, specifically thermo-plastic that can improve their mechanical and thermal properties. The study investigated the integration of other flame-retardant additives with APP, such as zinc borate, magnesium dehydrates, aluminium trihydrate and different polymeric systems, to promote the use of APP flame retardants for safer products.

2. Methodology

The Process of fabrication of intumescent fire retardant coating and its testing consisted of 5 steps.

2.1. Selection of Material

On the basis of background studies with respect to the role of material as fire-retardant, 7 different materials were considered for the fabrication of fire-retardant coatings. 1-Titanium dioxide (TiO_2), 2-Ammonium polyphosphate (APP), 3-Zinc borate, 4-Epoxy hardener, 5-Kaolinite (Clay), 6-Melamine (MEL) and 7-Expandable Graphite (EG) as shown
Table 1: List of Materials.



		
1-Epoxy Hardener	6- Zinc Borate	7- Kaolinite

Table 1: List of Materials

2.2. Fabrication of Coating

To achieve the most effective fire retardant coating, a combination of different materials was utilised to create five distinct formulations, each weighing a total of 200 grams. The quantities of the various materials differed in each formulation, resulting in different outcomes and providing insights into the properties of each component. The exact amount of each material was weighed using a top load balance, and added to a bowl along with the epoxy hardener. The mixture was then stirred with a mixer at 250 RPM for 5-7 min shown in Figure 1: Material Mixing through Shear Mixer. After that, the mixture was introduced by hardener, and it was stirred for another 3 minutes.

Table 2: Formulations of IFRC.

Samples	TiO_2	Epoxy	MEL	APP	Clay	EG	Hardner	ZB	Total
IFRC1	3	41.5	9	11	0	4	31.5	0	100%
IFRC2	3	31.6	9	11	5	4	37.4	0	100%
IFRC3	4.5	32.5	11	7	3.5	3.5	36.5	1.5	100%
IFRC4	4.5	31	11	5	3.5	7	36.5	1.5	100%
IFRC5	3	31.6	11	6	1.5	8	31.4	1.5	100%

Fig. 1: Material Mixing through Shear Mixer

2.2.1 Specimen Preparation

Mild steel was considered as a specimen, which contains less than 0.45% carbon, is strong and malleable but not easily tempered or hardened. Dimension of specimen was considered as follows Table 3: Mild Steel Specimen Parameters;

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a	L	4 inch
b	W	4.5 inch

c	T	2.5 mm

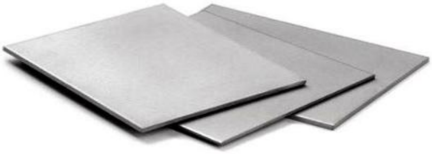

Fig. 2: Mild Steel Plates

2.2.2 Applying Coating

To apply 0.50 mm thickness IFR coating to mild steel plates, the prepared paste was subsequently poured in a frame with accurate dimensions of 4 inches by 4.5 inches and a height of 4.50 mm which made top edge of walls 0.50 mm up from the surface of plate. Smooth surface of IFRC was leveled by giving temperature through heat gun by setting temperature of 25 C of air which made viscosity of epoxy hardener low. At this stage coating paste maintained its coating surface. All Specimen with coating from IFRC 1 to IFRC 2.

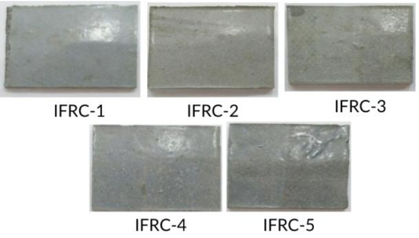

Fig. 3: Specimen with IFRC 1 -5

2.3. Testing

The plates were subjected to a Bunsen burner test after curing it for 48 hours. A basic gas-powered Bunsen burner was used to conduct the test at 650°C temperature to achieve the desired outcomes as shown below Figure. 4: Experimental Setup Bensen Gas Cutter. The conduct of the materials during the burning process was observed, as well as the behaviour and temperature of the specimen's reverse side. To ensure accurate findings, the temperature of the burning coated materials was noted every 15 seconds for a 5-minute period, and these readings were taken using a temperature gun.

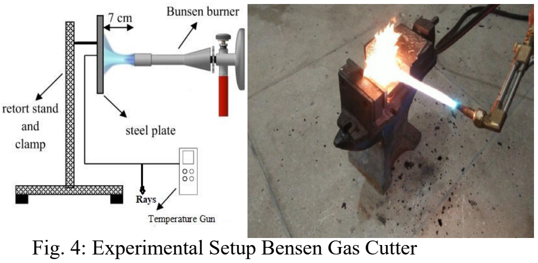

Fig. 4: Experimental Setup Bensen Gas Cutter

An infrared thermometer, commonly referred to as a temperature gun, is used to measure the temperature of the opposite side of the plate. Heat transfer rate was observed through temperature gun device shown in Figure 5: Temperature Gun Device.

Fig. 5: Temperature Gun Device

To evaluate the heat resistance capabilities of the mild steel plate coatings, they were exposed to a Bunsen burner flame at a distance of 7 cm, and the gas consumption rate was 170-190 g/h. The digital temperature gun was used to record the backside plate temperatures of the coated plates, which were measured every 15 seconds for 5 minutes as shown in Figure 6: After Test Specimen. The results were obtained by taking the average reading value of the digital temperature gun. To ensure data accuracy, the test was repeated three times for each formulation using three different mild steel plates of the same type.

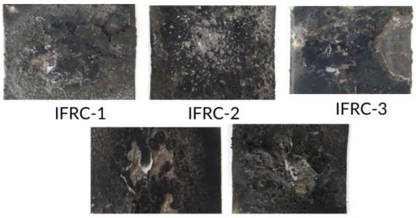

Fig. 6: After Test Specimen

The photographs of the coating after the Bunsen burner test are depicted. The intumescent coatings produce a dense carbon layer when exposed to fire, which acts as a barrier for heat transfer between the fire and the steel plate as show in Table 4: Temperature values of Specimen.

Table 4: Temperature values of Specimen

Specimen	Flame Temperature at Front side	Plate Temperature at Back side
	Mild Steel	Mild Steel
Simple Mild Steel	650 °C	460 °C
IFRC1	650 °C	162 °C
IFRC2	650 °C	155 °C
IFRC3	650 °C	135 °C
IFRC4	650 °C	117.4 °C
IFRC5	650 °C	136 °C

3. Results and Discussion

The above table summarises the front side flame temperature and the opposite side plate temperature of the samples. Simple mild steel had the highest back side plate temperature, whereas the intumescent coatings had significantly lower back side plate temperatures. The IFRC1 formulation had the highest back side plate temperature among the coatings, with a temperature of 162 °C. In comparison, the IFRC4 coating had the lowest temperature, with a back side plate temperature of only 117.4 °C. Overall, these results suggest that the intumescent coatings can act as an effective heat transfer barrier between fire and the steel plate as shown in Figure 7: Time Temperature Profile of Specimen.

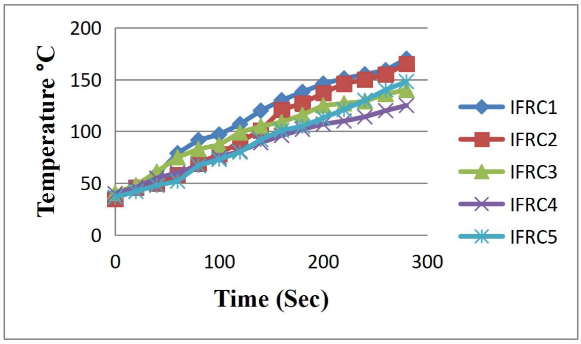

Fig. 7: Time Temperature Profile of Specimen

Table 5: Expansion of Specimen Coatings in layers

Samples	Thickness After Coating	Thickness After Testing
	Mild Steel	Mild Steel
IFRC1	3.00mm	8.8mm
IFRC2	3.02mm	7.4mm
IFRC3	3.03mm	7.2mm
IFRC4	3.01mm	7.5mm
IFRC5	3.05mm	9mm

Figure 7 displays the time-temperature profiles of mild steel without coating, which illustrate the effectiveness of coating in reducing the backside temperature of plates. Among the different formulations investigated, IFRC1 exhibited the highest temperature of 172.5°C at the back side of mild plates and was with low stability but strong bond as compared to the other formulations, mainly because it lacked clay. This underlines the significance of clay as an insulating and stable material during firing tests.

In contrast, IFRC2 demonstrated better stability and a lower back side temperature of 165°C compared to IFRC1, due to the excessive presence of TiO_2 and clay in the formulation. IFRC3 contained zinc borate and TiO_2, which were not present in the previous two coatings, resulting in a lower back side temperature than other two formulations i-e IFRC1 and IFRC2.

The Formulation IFRC-4 had the similar formulation as IFRC-3 but with the addition of EG in a larger proportion. Its temperature at the back side was 137.5°C, indicating that the surface that burnt still acted as a flame penetration/fire retardant. The Formulation IFRC-5 had higher amount of EG, low amount of clay, and TiO_2 than formulation IFRC-4. The functionality of this formulation indicated that the coating expanded because of excessive amounts of EG as shown in Table 5, resulting in a temperature backside of the mild plate that fell between formulation IFRC-3 and formulation IFRC-3.

4. Conclusion

This study aims to develop and assess intumescent fire retardant coatings by integrating Seven different materials as discussed above. These substances were mixed in a material mixer at a speed of 200 rpm and applied to mild steel plates and pinewood pieces. The coatings were then subjected to a Bunsen burner test using a standard experimental setup for analysis. The findings demonstrated that

1- IFRC4 was the most effective formulation, with a temperature of 127.5°C and consisting of 27.5% Epoxy Hardener, 4.5% Clay, 10% MEL, 30% Epoxy resin, 3.5% TiO_2, 6% EG, 2.5% Zinc borate,and 6% APPby weight.

2- Due to the double proportion of EG compared to IFRC3, the IFRC4 coating remained stable and firmly bonded at a flame temperature of 700°C for over five minutes.

3- The EG in IFRC4 expanded during a fire and was demonstrated to resist fire penetration and spread experimentally.

4- The average backside temperature of mild steel coated with IFRC4 was calculated at 127.5°C, indicating the lowest rate of heat transfer in comparison to the other coatings.

5- IFRC4 expanded four times its initial coating thickness after the Bunsen burner test, whereas IFRC1, with the highest backside plate temperature of 172.5°C, expanded more than IFRC4 due to its ingredients.

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