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Fire Resistance of Glass Wool and Rockwool Hybrid Fibre Reinforced Intumescent Coating

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Abstract – *Intumescent coating is widely used in industry for protecting steel from fire or high temperature. However, basic intumescent coating cannot protect the substrate effectively since its char is soft and easily penetrated by fire. Therefore, the purpose of this study is to examine the effects of several hybrid fibre's loading combinations and lengths to the coating's fire resistance. Glass wool and Rockwool fibres were selected as the reinforcements. A reference intumescent coating was developed with materials such as APP, PER, MEL and boric acid, mixed with BPA epoxy and PA. Coating thickness was approximately 2.65mm. Coating's thermal stability and residual weight were examined using TGA. Char expansions were measured upon exposure to electric fire furnace test. Fire resistance of the coatings for 40 minutes duration were determined from 1000°C Bunsen burner test. The research concluded that long fire retardant fibres improved fire resistance of intumescent coating; with the formula having single Rockwool fibre showed better performance than other combinations. In addition, hybrid fibre reinforcement with greater Rockwool fibre loading had increase char expansion and residual weight of the coating.*

Keywords: Intumescent coating, Fire resistance, Hybrid fibre, TGA

1.0 INTRODUCTION

For many years, fire protecting system has been a mandatory for commercial buildings. The application of intumescent coatings as fire protection for different kind of base materials is skyrocketing throughout the world. This is because most of the materials widely used in the constructions are unable to withstand high temperatures. Steel for example will lose significant amount of its load bearing capability at elevated temperature. An intumescent coating is an effective fire protection with two main benefits that are prevention of heat transfer to the substrate material and retardation of flame spread. Furthermore, it does not change the original properties of substrate materials. In the event of fire, it reacts and expands into a thick char layer of low thermal conductivity to insulate and protect the base material.

Thick film epoxy-based intumescent coatings are widely used in offshore installations and industrial facilities to offer fire protection to steelwork against hydrocarbon pool and jet fires, as well as offering corrosion protection [1]. Additionally, in oil and gas industry, the fire protection coatings must resist highly erosive forces from ignited pressurised gases, as well as comparatively high imposed heat fluxes coming from fire incident [2]. The thickness of these coatings ranges between 5 and 25 mm, which relies on the required fire resistance, the steel

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section type and section factor (the ratio of heated surface area to volume of steel) and the limiting temperature, typically based on design code requirements [3].

Intumescent coatings made of only three basic ingredients i.e. char-forming material, a catalyst, a blowing agent and binders [4] usually produce soft char, due to its lacking of strength [5, 8]. This affects the efficiency of the coating in protecting the base structure when exposed to high temperature. The char layer can be depleted by physical erosion and chemical processes such as oxidation thus, its protection capability is reduced. The depletion causes it to crumble and without sufficient strength, a char layer will easily falling off thus fail to protect substrate material. Commonly, the coatings are applied onto substrate materials together with external reinforcing materials such as carbon mesh, wire mesh and etc. to maintain its adhesion and integrity in the event of fire [2, 6].

The objectives of this paper are to develop and synthesize hybrid fibre reinforced intumescent coating using glass wool and Rockwool fibres. Significant contribution of the paper is the documentation of the effects of fibre length and fibre loading towards fire resistance of intumescent coatings.

There are several studies about the effects of using individual fibres such as glass wool, Rockwool and ceramic wool fibres to increase fire performance of intumescent coating [6, 8]. Glass wool is an insulating material made from fibres of glass arranged using a binder. It obtains thermal insulation properties by trapping numerous small pockets of air between the glasses during its manufacturing process. Glass wool fibre is a thermal insulation of intertwined and flexible glass fibres, which causes it to "package" air, resulting in a low density, which is varied through compression and binder content. Stone wool fibre is a furnace product of spinning or drawing molten rock at temperature of about 1600°C. The final product is a mass of fine, intertwined fibres with a typical diameter of 6 to 10 µm. Rockwool fibre mat expands nine times its original thickness, forming thick layer that is resistant to fire and erosion [9].

2.0 METHODOLOGY

2.1 Intumescent Coating Formulations and Hybrid Fibres

In this research, eleven epoxy-based intumescent coating formulations with varying fibre contents and length have been developed. They consist of one unreinforced formula, four single fibre reinforced formulations and six hybrid fibre reinforced formulations as presented in Table 1. The samples are labelled based on their fibre length and content. A sample without fibre content is labelled as C1. Fig. 1 shows the guide for sample labelling.

Seven materials are used such as Bisphenol A epoxy resin (BPA) and polyamide amine (PA) by Mc-Growth Chemical, ammonium polyphosphate (APP) by Clariant, melamine (MEL) by SABIC, boric acid (BA) and pentaerythritol (PER) by MERCK, and glass wool (GW) and Rockwool (RW) fibres by FOSTER. Powder materials are first grinded using Panasonic mixer grinder. The materials are then mixed with hybrid fibre and liquid ingredients using Caframo mixer with straight blade impeller at speed 30-40rpm until a homogenous solution with hybrid fibres evenly dispersed, is formed.

For each steel substrate, 20 g of coating are applied, forming an average thickness of about 2.65 mm. The coatings are then, left to dry at room temperature. Measurement of coating

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thickness is done using a vernier caliper. Average dry coating thickness for a single sample is the average from four measurements at each side of the coating. Fig. 2 shows two-sizes of steel plate coated with brownish coloured, hybrid fibre reinforced intumescent coating.

Table 1: Intumescent coating formulations for present research in weight %

No	Label	BPA (Epoxy) (%)	PA (Hardener) (%)	APP (%)	PER (%)	MEL (%)	BA (%)	GW Fibre (%)	RW Fibre (%)	Fibre Length (mm)
1	C1	44.4	22.2	11.1	5.6	11.1	5.6	0	0	0
2	SG10	38.4	18.2	11.1	5.6	11.1	5.6	10	0	6
3	SR10	38.4	18.2	11.1	5.6	11.1	5.6	0	10	6
4	SG7R3	38.4	18.2	11.1	5.6	11.1	5.6	7	3	6
5	SG3R7	38.4	18.2	11.1	5.6	11.1	5.6	3	7	6
6	SG5R5	38.4	18.2	11.1	5.6	11.1	5.6	5	5	6
7	LG10	38.4	18.2	11.1	5.6	11.1	5.6	10	0	12
8	LR10	38.4	18.2	11.1	5.6	11.1	5.6	0	10	12
9	LG7R3	38.4	18.2	11.1	5.6	11.1	5.6	7	3	12
10	LG3R7	38.4	18.2	11.1	5.6	11.1	5.6	3	7	12
11	LG5R5	38.4	18.2	11.1	5.6	11.1	5.6	5	5	12

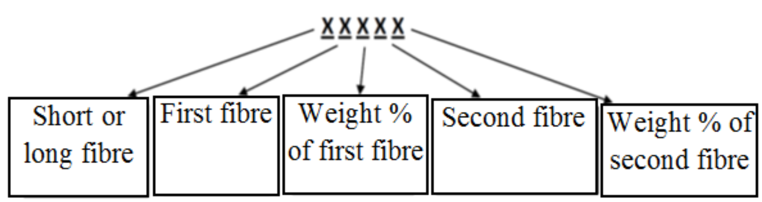

Figure 1: Sample labelling guide

2.2 Intumescent Coating's Char Expansion Test

Furnace test allows free char expansion of the coating when exposed to high temperature in a closed environment. General rule of thumb is, the higher the expansion indicates the better is the coating's fire protection. For the test, fully dried coating samples are placed in an electric furnace and furnace temperature is raised from room temperature to 400°C in 1 hour and the temperature is maintained for about 10 minutes. Next, temperature is lowered to 30°C in 1 hour and the samples are left for cooling for about 10 hours. Then, thickness of each charred sample is measured.

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2.3 Thermal Stability Test

Thermogravimetric analysis (TGA) is the thermal analysis technique used to measure changes in physical and chemical properties of coatings as a function of increasing temperature. The flow rate of the inert, oxidative gas was 20 mL/min. The starting temperature was 30°C and reached maximum temperature of 790°C with heat rate of 20°C/min. The gas environment was thermal-oxidative combination.

2.4 Fire Resistance Test

Bunsen burner test was used to evaluate fire resistance performance of the coating. The method involved recording the temperature at the bare back of the coated steel using K-type thermocouple linked to a data logger. The flame is targeted to the centre of the vertically positioned sample. The distance between the plate and Bunsen burner was fixed to 7 cm and the samples were exposed to a fire horizontally, which reached 1000°C in 8 minutes for 40 minutes. Experiments were carried out in duplicate to ensure repeatable results.

(a) Close-up of the coating.

(b) 5 cm X 5 cm (left group) and 10 cm X 10 cm (right group) samples.

Figure 2: Brownish coloured intumescent coating with different hybrid fibre configuration as coated onto steel samples as in (a) and (b) on the left for furnace fire and right for Bunsen fire

3.0 RESULTS AND DISCUSSION

3.1 Effects of Hybrid Fibres to Char Expansion

Intumescent coating needs sufficient swollen char to effectively act as barrier to insulate the steel from adverse effects of flame and heat. Free expansion results of the coatings are shown in Table 2. All of the fibre reinforced coatings except for LG7R3 expanded more than the unreinforced coating, C1 as shown in Fig. 3.

The highest expansion was recorded by sample SG10 followed by SG5R5 and LG3R7. Sample SG10 recorded an expansion of 3.48 mm (134.9%), SG5R5 with 3.02 mm (102%) and LG3R7 with 2.56 mm (95.5%). The black-coloured chars were observed to have rough surface while sufficiently covered and adhered to the steel substrates as evident in Fig. 4.

It was found that samples with shorter fibre length developed higher char expansion if compared to the samples with longer fibre length. As an example at the same fibre loading of

10 wt.%, intumescent coating reinforced with glass wool fibres only at 6 mm, SG10 swell 58% more than LG10, the sample reinforced with fibres doubled the length at 12 mm. This showed that the shorter GW fibre length in the coating formula resulted in greater char expansion indicating increase in fire protection.

Table 2: Intumescent coating formulations from 400°C furnace fire

No	Sample	Before Test Thickness (mm)	After Test Thickness (mm)	Expansion (difference, mm)	Expansion (%)
1	C1	2.06	3.20	1.14	55.3
2	SG10	2.58	6.06	3.48	134.9
3	SR10	2.98	5.24	2.26	75.8
4	SG7R3	3.00	5.02	2.02	67.3
5	SG3R7	2.30	5.46	3.16	92.5
6	SG5R5	2.96	5.98	3.02	102.0
7	LG10	2.68	4.74	2.06	76.9
8	LR10	2.68	4.72	2.04	76.1
9	LG7R3	2.50	3.46	0.96	38.4
10	LG3R7	2.68	5.24	2.56	95.5
11	LG5R5	2.70	4.58	1.88	69.6

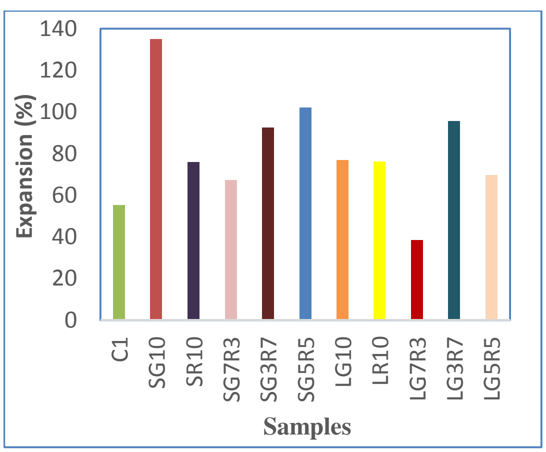

Figure 3: Char expansion in percentage for each coating sample

Figure 4: The black char fully covered the 5 cm X 5 cm steel plates

Contrarily however, slightly higher char height was recorded for coating reinforced with longer single RW fibre than the shorter ones at only 0.3%. This may be due to the fact that the average diameter of GW fibre is about four times that of RW fibre, while the latter is four times denser than the former. Longer fibre always has larger aspect ratio than the shorter one.

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The coatings formulated with hybrid fibre, also showed different trend in char expansion between short fibre and long fibre. For coatings with short hybrid fibres, highest expansion was recorded by SG5R5, where fibre loading was equal between GW and RW fibres. On the other hand, for long hybrid fibres samples, LG3R7 with 3 wt.% GW and 7 wt.% RW fibres resulted in highest char expansion, which was 25.9% greater than LG5R5.

For both fibre-length groups, intumescent coatings (SG7R3 and LG7R3) with more GW fibres i.e. 7 wt.%, if compared to RW fibres at 3 wt.% yielded the lowest char growth. This shows that intumescent char expansion is affected by the aspect ratio of the hybrid fibres. Due to its smaller diameter and hence larger aspect ratio, addition of 50% to 70% RW fibres over GW fibres created more opportunity for the char growth.

3.2 Effects of Hybrid Fibre to Residual Weight of the Coating

The residual weight as plotted against temperature as a result of TGA analysis explained the degradation experienced by coating samples. Generally, a sample that record a higher residual weight would offer better fire resistance and have a better thermal stability. Fig. 5 and 6 show the results for two short fibre reinforced samples, SG3R7 (3% GW fibre 7% RW fibre) and SR10 (10% RW fibre). The weight loss of the coatings was 2%, respectively at the beginning of the experiments, mostly due to fraction of substances and resin decomposed, while volatile materials vaporized. Similar finding was also reported by Gu et al. [10].

In general, both curves showed similar pattern because the changes in fibre content was small to produce obvious deviation in thermal stability. As reported in our previous research [6], in general, there are three distinctive decomposition temperatures; (1) by moisture removal and decomposition of low temperature polymeric materials, (2) at approximately 250°C by the three key intumescent ingredients and, (3) at 500°C for the main intumescence reaction.

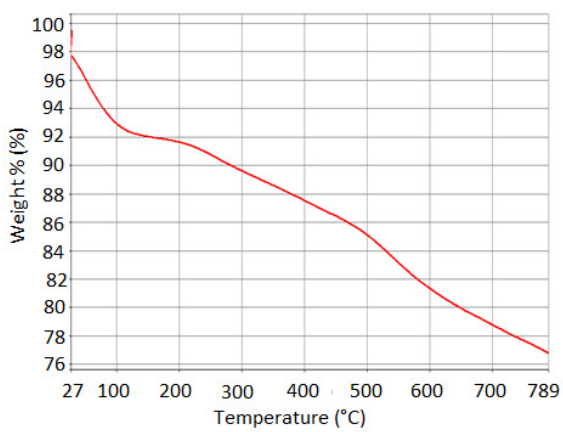

Figure 5: TGA curve for SG3R7

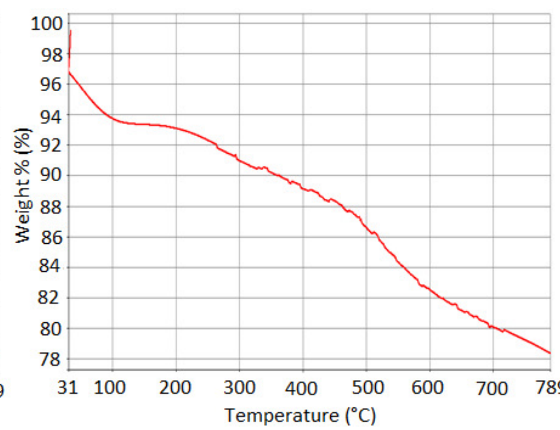

Figure 6: TGA curve for SR10

In the current research, residual weights at 750°C obtained from samples SG3R7 and SR10 were 77.1 wt.% and 79.2 wt.%, respectively. These results when compared to unreinforced formula (C1), the increment in residual weight were by 27.2% and 30.7%, respectively. For comparison, another study recorded residual weight for three samples (X, Y and Z) at only 28.07 wt.%, 27.70 wt. % and 26.98 wt.%, respectively [7]. At 790°C, the results showed that

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samples SG3R7 produced 76 wt.% residue, and SR10 is left with 78 wt.% residual weight, which are 35.7% and 39.3% greater than C1.

Previous studies stated that there should be high amount of residue left at 800°C temperature or higher in order for the coatings to effectively protect the steel [6-8]. A sample with high amount of residual weight indicates a better homogenous char. This high amount of residual limited heat transfer to the substrate and reduced the gases feeding combustion process.

Based on the results, the sample that had the highest residual weight was the sample reinforced with 10% of single Rockwool fibre. Results also showed that residual weight increased due to increasing of composition percentage of Rockwool fibre that provided a better thermal stability for the coating and thus greater fire protection for the steel substrate.

3.3 Effects of Hybrid Fibre to Intumescent Coating's Fire Resistance

Effects of long-hybrid fibre reinforcement to fire resistance performance of intumescent coating are further studied using direct forced fire. Six samples were selected for this study; unreinforced (C1), single fibre (LG10 and LR10) and hybrid fibre (LG3R7, LG5R5, LG7R3). The results are shown in Fig. 7, while the physical of the burnt samples are shown in Fig. 8. Sample LR10, with 10 wt.% RW fibre recorded the lowest temperature at the end of the test, at 74.5°C. This is followed by another single fibre formulation, LG10 at temperature, 82.2°C.

The best fire protection by hybrid fibre coatings was given by LG3R7 at 95.1°C and followed by LG5R5 (96.9°C) and LG7R3 (105.6°C). As expected, the unreinforced sample C1 recorded the highest steel temperature at 136.7°C. These results are not surprising because RW being smaller in size compared to GW possess more surface area to resist fire attack. The content of RW in the intumescent coatings helped to increase thermal insulation properties of the coatings and avoid detrimental effects to integrity of steel structures by limiting its temperature to be well below the critical temperature [6-8].

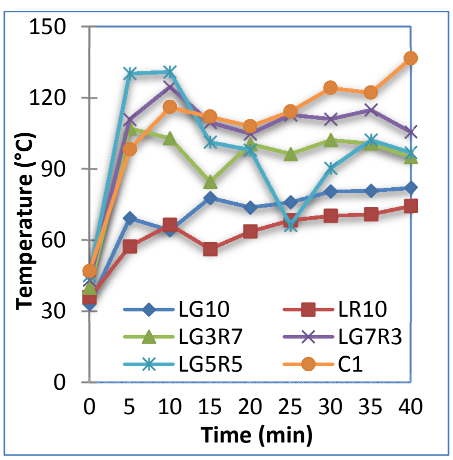

Figure 7: Fire resistance curves for various intumescent samples

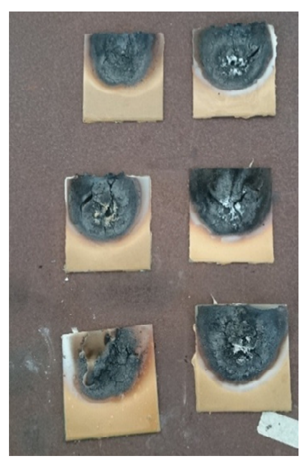

Figure 8: Intumescent coated steel, 10 cm X 10 cm samples after fire resistance test

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4.0 CONCLUSSION

This research studied the effects of hybrid fibre length and hybrid fibre loading to fire protection characteristics of intumescent coating. Eleven formulations were prepared; unreinforced, single fibre reinforced and hybrid fibre reinforced coatings. Glass wool fibre and Rockwool fibre were selected for the preparation of hybrid fibre reinforced formulations. Two fibre lengths were chosen; short fibre at 6 mm and long fibre at 12 mm. Fibre loading was kept at 10 wt.%, for fibre reinforced coatings with each fibre contributed either 0 wt.%, 3 wt.%, 7 wt.% or 10 wt.%. In conclusion, fibre reinforced intumescent coatings performed better thermal and fire protection than the unreinforced coating. Single fibre reinforced formulas had shown better performance than the hybrid fibre coatings

Both single and hybrid GW and RW fibres are found to promote char expansion. Due to its light in density, short–single- glass wool fibre reinforced formulation, gave out the highest char expansion. Better thermal stability in the form of higher residual weight after fire is the effect of higher fibre loading. For the case of hybrid fibre, increased in RW fibre loading, which is a more fire retardant fibre than GW fibre resulted better fire protection. Furthermore, short fibres may have produced better fibre dispersion in the coating and therefore performed better in fire to retain higher residue. Finally, a higher density (than GW) long-single-Rockwool fibre reinforced intumescent coating provided the greatest fire resistance to steel.

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