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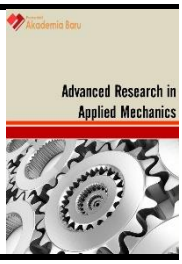

Optimization of Flame-Retardant Additives on Fire Protection Performance and Thermal Properties of Water-Based Intumescent Coating

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ABSTRACT

The aim of this research project was to optimize the effect of flame-retardant additives on the fire protection performances and the thermal properties of water-based intumescent fire protective coatings on steel structures. Four intumescent coating formulations with different ratios of flame-retardant additives (Ammonium polyphosphate (APP), Melamine (MEL) and Pentaerythritol (PER)) were formulated and mixed with the flame-retardant fillers and the water-based polymer binder. The coatings were characterized and examined by using the Bunsen burner test, static immersion test, furnace test, adhesion test, freeze-thaw cycle test, scanning electron microscopy (SEM) and Thermogravimetri Analysis (TGA). The results showed that the Coating 1 with the ratio of APP: MEL: PER of 2:1:1 was effective in fire protection performance, with good quality of water resistance, adhesion strength, promoting a better uniform char layer formation and thermal stability. Significantly, the optimized flame-retardant additives have proved to be efficient in the protection of steel structures against fire and weather resistance.

1. Introduction

In recent years, there has been a notable rise in demand for intumescent coatings used on structural steelwork, including columns, girders and frameworks, along with castellated and complex steel building elements. Steel begins to lose its structural properties above 500 ºC in case of fire and tends to distort, leading to the collapse of building structures. Indeed, the prevention of the collapse of the steel structures is of paramount importance to ensure the time for safe evacuation from burning buildings, and it represents a primary requirement of building regulations in many countries [1]. The use of intumescent coatings is one of the most efficient ways to protect different substrates against fire. The expansion process of intumescent coating is caused by the interaction of three

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precisely formulated flame-retardant components: (1) acid source (ammonium polyphosphate (APP)), (2) carbon source (pentaerythritol (PER)) and (3) expanding agent (melamine (MEL)) [2-4]. The use of flame retardant ingredients may avoid a small fire from flattering a major disaster. During the intumescent process, the binder becomes important due to two significant effects: it contributes to the char layer expansion and ensures the formation of uniform char foam structure [5,6]. Several advantages of using intumescent coating over other methods of structural fire protection are the aesthetically pleasing finish it gives to steelwork, fast application, easy to cover complex details and maintaining the intrinsic properties of steel structures [7].

Basically, there are two types of intumescent coatings. There are solvent-based and water-based intumescent coatings. This experimental work focuses on the investigation of thermal properties and fire protection of water-based intumescent coating. This coating is a user friendly alternative to solvent-based as it has a low toxicity and promoting more environmentally friendly. During a fire, when the temperature rises between 280 and 350 ˚C, the development of intumescence occurs by decomposing of coating (melt zone) [8]. As the temperature continue to increase up to 350 to 420 ˚C, the degradation process of the intumescent coating takes place (reaction zone). Above to 420 ˚C, it will lead to a formation of a layer of carbonaceous species, a porous char layer with excellent heat insulation to prevent it from entering steel lie underneath it for 1 to 3 hours (charring zone) [9]. This provides sufficient time for evacuation and hence save more lives. The optimization of the formulation is important to form an effective char layer with high durability and uniform foam structure to protect the substrates [10].

This research project is mainly focused on the effects of different composition of flame-retardant additives on the fire protection performance and thermal properties of water-based intumescent coating. Over a few decades, intumescent flame-retardants are commonly used in the field of fire protective coatings because they demonstrated an excellent flame-retarding performance when applied on different types of materials like steel, wood, cables and even polymer. The flame-retardant additives play a significant role in forming the light char layer which possesses a very low heat conductivity to prevent the spread of fire to the substrate. Besides that, filler is needed to strengthen the char layer structure coating by including resistance of fire ignition, reducing the rate of expansion of char layer and minimizing gases emission. This is because char layer formed exhibit poor adhesion strength and very easy to oxidize. Therefore, fire-retardant fillers are needed to enhance the performance of the coating in many aspects. There are a lot of fire retardant fillers which are widely used including the titanium dioxide, aluminium hydroxide, magnesium hydroxide and calcium carbonate [11]. Binders are important in supporting the formed char and prevent it from collapsing. It also helps in softening and charring during a fire. This project studies the influences of composition of flame-retardant additives on fire protective performance and mechanical properties of a water-based intumescent coating during an event of fire. The intumescent coatings with different formulations of flame retardant additives will be investigated through several experiments such as Bunsen burner test, static immersion test, furnace test, adhesion test, thermogravimetric analysis, scanning electron microscopy and freeze-thaw cycle test.

2. Materials and Experimental

Intumescent coating was prepared using water-based vinyl acetate acrylic copolymer, flame-retardant fillers and flame-retardant additives. All these ingredients were mixed by using high speed disperse mixer. The compositions of all the materials were shown in Table 1. It shown those flame-retardant additives were the only changing variable whereas the binder and fillers are kept constant.

Each formulation was made up of 100 wt.% of ingredient. Polymer binder, flame-retardant additives and fillers consist 50 wt.%, 40 wt.% and 10 wt.%, respectively.

Table 1
Composition of intumescent coating formulations

Coating sample	*Vinyl acetate acrylic copolymer*	Composition (wt.%)			
		Flame-retardant fillers	Flame-retardant additives		
		$TiO_2/CaCO_3/Al(OH)_3/CaSiO_3$/EG	APP	PER	MEL
1	50	3/2/2/2/1	20	10	10
2	50	3/2/2/2/1	24	8	8
3	*50*	3/2/2/2/1	13.25	13.25	13.25
4	50	3/2/2/2/1	10	20	10

Figure 1 shows the technique measurements that have been conducted in order to study the properties of water-based intumescent coating. Firstly, the purpose of furnace test is to investigate the thickness of char layer formed by each formulation when it was heated under 400 ˚C, 500 ˚C and 600 ˚C. Bunsen burner test was used to examine the performance of intumescent coating. Besides that, mechanical properties of the coatings were tested using adhesion strength test and static immersion test. TGA was used to investigate the thermal degradation of coating and SEM was conducted to examine the morphology of char layers.

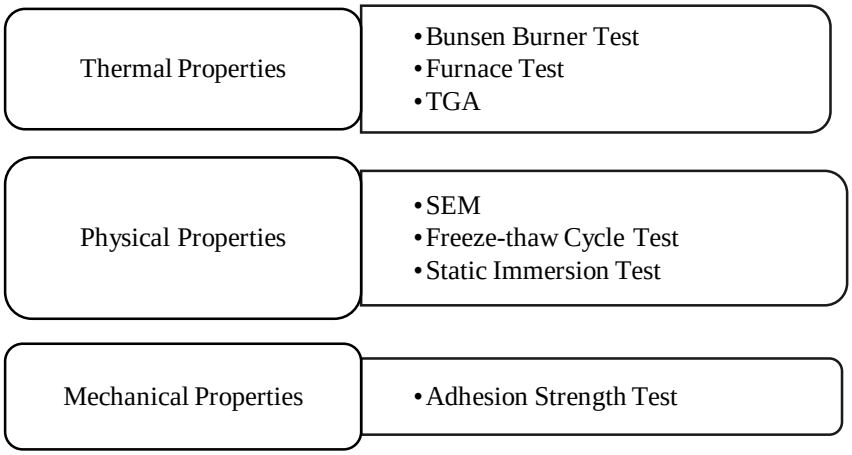

Fig. 1. Characterizations of intumescent coating samples

2.1 Bunsen Burner Test

Bunsen burner test was conducted to investigate the fire protection performance of different coating formulations. The coating formulations were applied on steel plates with dimensions of 100 mm x 100 mm x 3 mm and thickness of 2.0 ±0.2 mm. The steel plate is then exposed to high temperature flame (about 1000˚C) for 60 min for each formulation. Besides that, the distance between steel plate and the Bunsen burner is fixed at about 8 cm to standardize the fire test. The change in temperature of the steel plates was recorded every minute. The thickness of char layer formed at the end of the experiment is observed and measured. Figure 2 shows the experimental set-up of Bunsen burner test.

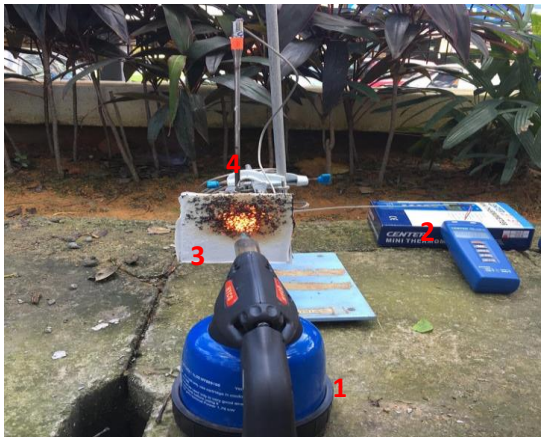

Fig. 2. Bunsen burner test setup (1) Bunsen burner, (2) digital thermometer, (3) sample and (4) thermocouple plate

2.2 Furnace Test

Furnace test was conducted to measure and observe the thickness of char layer formed at temperatures of 400˚C, 500˚C and 600˚C, respectively. Coating was coated on 50 mm x 50 mm x 2 mm steel plate with a thickness of 2.0 ±0.2 mm and left to dry for 1 week before conducting the furnace test. The furnace test was carried out on 5 steel plates coated with four different formulations.

2.3 Static Immersion Test

Static immersion test is a typical technique that investigates the water resistance of thin film such as intumescent coating using the gravimetric method. Coating was poured into plastic mould with thickness of 2 mm and left it to dry for a week. After the coating sample is completely dried, it is being removed from the mould and placed into a plastic container that filled with water. Each coating sample is removed from water after time interval of three days and the excess water on the surfaces is removed by using a piece of tissue paper. Then, the weight of sample before and after immersed in water is weighed using a weighing machine and the results are recorded. Weight change of the coating is recorded daily for consecutive two weeks and water uptake ratios for each sample were calculated using Eq.1.

$$E_{sw}\ (\%) = [(W_e - W_o)/W_o] \times 100\% \quad (1)$$

where, E_{sw} = the water uptake ratio of film, %
W_e = the weight of intumescent coating before water immersion, g
W_o = the dry weight of the intumescent coating after water immersion

2.4 Scanning Electron Microscope (SEM)

Studies of the morphology of char layer formed were carried out using a scanning electron microscope (SEM) by emitting electron and receiving the electron to form an image. SEM is operated at low beam energy of 1Kv to protect and prevent thermal damage on the char layer. SEM is operated

using two level of magnification which are 4000 and 8000 magnifications. After the Bunsen burner test, a small piece of char layer was taken from the centre of each coating sample for SEM test.

2.5 Thermogravimetric Analysis (TGA)

TGA was conducted to investigate the thermal degradation of intumescent coatings. About 5-8 mg of thin film was placed in a ceramic crucible with a heating rate of 10˚C per minute under the airflow.

2.6 Adhesion Strength Test

The adhesion strength test was conducted to determine the bonding strength of each coating formulations. The cylindrical rods of same dimension is prepared and being polished with sand paper to remove impurities on the surface. Then, the coating thickness of 2 ±0.2 mm was applied on the top round surface of the cylindrical rod and left aside for about 4 days for it to dry completely. After the coating was dried completely, another same dimension of cylindrical rod is attached onto the coating with epoxy glue of 1.0 ±0.2mm and then left it to dry again. The Instron microtester was used to carry out the adhesion test. The force that used to break the coating was recorded and the adhesion strength for each coating samples are calculated using the Eq. 2 below:

$$F_b = \frac{F}{A} \quad (2)$$

where, F_b=Bonding strength (N/m^2)

F = Crack charge (N)

A= Surface area of intumescent coating on the top round surface

2.7 Freeze-Thaw Cycle Test

The purpose of freeze-thaw cycle test is to determine and investigate the weather resistance of different formulation of intumescent coatings. All the steel plates were polished with sand paper before the coating was applied. Intumescent coatings were coated on 50 mm x 70 mm x 1 mm steel plates with thickness of 2.0 ±0.2 mm and left aside for about 1 week for it to completely dry. After 1 week, the steel plates coated with coatings were placed into the freezer at the temperature of -20˚C for 8 hours, and then the same samples were left in room temperature for 8 hours before placing into the drying oven (50˚C) for another 8 hours continuously. This process throughout the test is recorded as freeze-thaw cycle period.

3. Results and Discussion

3.1 Bunsen Burner Test

For Bunsen burner test, all the four coating samples were heated for 60 minutes and the temperature profiles during exposure to fire has recorded using a digital hand-held thermometer. Moreover, the thickness of the char layer after burning has measured in order to check the fire resistive performance of the intumescent coating. Figure 3 showed the temperature profiles of coating samples after 60 minutes fire. During the heating process, coating sample would eventually reach its equilibrium temperature and maximum thickness of char layer. The equilibrium

temperature and thickness of char layer formed are used to indicate the fire protection performance of coating samples.

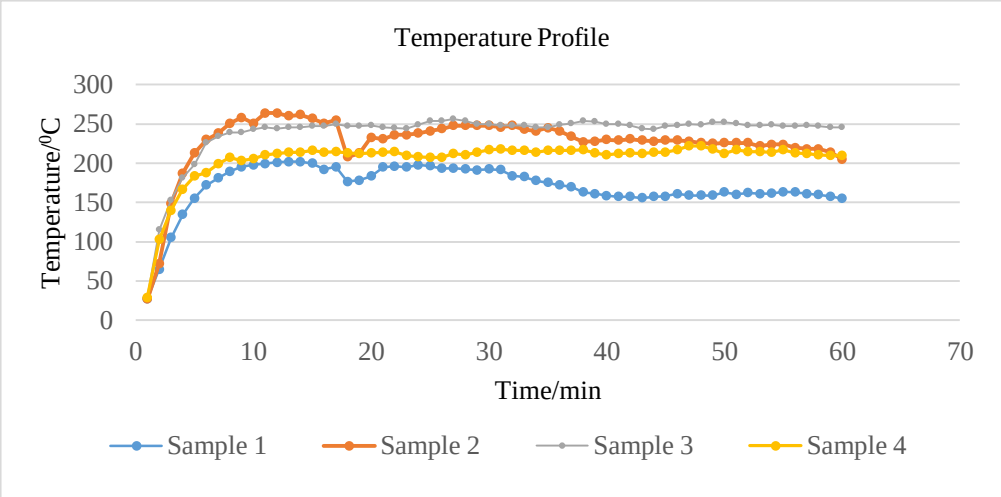

Fig. 3. Temperature profile of all samples after the Bunsen burner test

Table 2
Equilibrium temperature and thickness of char layer of coating samples

Coating samples	Equilibrium temperature (˚C)	Thickness of char layer formed (mm)
1	157	16.5
2	219	14.0
3	247	18.0
4	226	16.0

The coating sample 1 showed the lowest equilibrium temperature which is 157˚C and the thickness of char layer is about 16 mm as shown in Table 2. In contrast, coating sample 3 has the highest equilibrium temperature of 247˚C. All of the four coating samples showed similar growth in temperature in the first 10 minutes of heating and then it reached equilibrium in temperature and remains unchanged for a certain period. By comparing in terms of equilibrium temperature, Coating 1 has the best fire protection performance as it has the lowest equilibrium temperature (157˚C) followed by, Coating 2 (219˚C), Coating 4 (226˚C) and Coating 3 (247˚C). Coating 1 which made up of APP, PER and MEL in the ratio of 2:1:1 had the lowest equilibrium temperature due to the formation of a more stable microstructure of char layer. This happened is mostly because of the addition of Ammonium Polyphosphate (APP) has enhanced the formation of mineral acid for the dehydration of carbonization source (PER) to occur. Therefore, it formed a more stable char layer compared to the other formulations. Effectives char protection that reduced the heat transfer to the substrate. In contrast, Coating 3 showed the worst fire protection performances even though it had good expansion of char layer (about 18.0 mm). This is due to the weak and porous char layer formed that unable to reduce the heat transfer to the steel plate effectively. Coatings 1 and 2 with higher ratio of APP showed a better fire protection as it had the lower equilibrium temperature compared to Coatings 3 and 4. This is because the extra APP promotes the formation of stronger bonding to protect the heat from reaching the substrate.

Besides the equilibrium temperature, the thickness of char layer formed was also being observed and measured. When the temperature increase, APP had released the amino acid that resulting in the decarbonation of CES and caused the expansion of the char layer. The char layer formed is crucial in preventing the fire spread and reaches the steel plate. In comparison, Coating 3 formed the

thickest char layer (18.0 mm) followed by Coating 1 (16.5 mm), Coating 4 (16.0 mm) and lastly Coating 2 (14.0 mm). Based on the result obtained, Coating 3 had thickest char layer does not exhibit the lowest equilibrium temperature. Therefore, it can be concluded that the fire protective performance does not depend solely on the thickness of char layer formed. Furthermore, Coating 3 provides the weakest fire protection because it has an unstable adhesion to the steel plate. During the heating process, some part of the char layer of Coating 3 falls off from the steel plate. This happened because of the weak bonding strength that failed to form a protective barrier for the steel plate. Therefore, the suitable composition of flame retardant additives allowed continuous adhesion between the coating and steel plate and yet providing longer-lasting protection.

3.2 Furnace Test

In furnace test, four samples with different compositions of flame retardant additives is heated under the temperature of 400˚C, 500˚C and 600˚C and the thickness of the char layer formed is measured and recorded. Based on the graph plotted in Figure 4, it shows that sample 3 formed the thickest char layer as compared to samples 1, 2 and 4 which indicated that Coating 3 had the best char layer expansion. Sample 3 reached 15 mm of char thickness at 600˚C. Sample 3 are composed of flame retardant additives which is APP, PER and MEL in the ratio of 1:1:1. This shows that the incorporation of same amount of flame-retardant additives into coating formulation resulted in best char layer expansion. Meanwhile, sample 2 formed thinnest char layer which is about 2 mm with the flame retardant additives ratio of 3:1:1. This indicates that excessive amount of ammonia polyphosphate may inhibits the expansion of char layer. Hence, it can be deduced that too much APP could restrict the formation of char layer. Therefore, optimization of the ratio of APP, MEL and PER plays a significant role in providing the best fire protective performance.

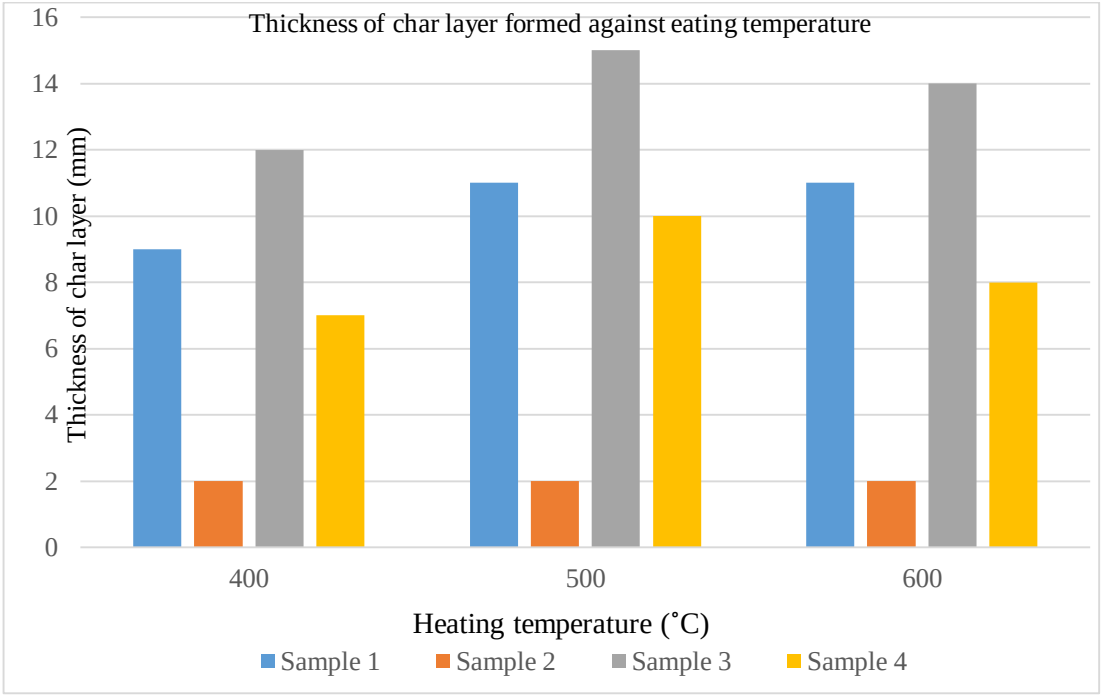

Fig. 4. Thickness of char layer formed by coating samples

3.3 Static Immersion Test

In static immersion test, four different coating formulations were placed into distilled water for a period of time and the weight before and after immersion was measured and recorded. During immersion test, there are basically two types of reactions that occurred on the coating which is migration and permeation. Permeation is a process of water molecules diffuse into coatings and causes the increase in weight of the coating, whereas migration is the process where coating particles moving out from the coating into the water and resulted in loses of weight. Figure 5 shows the water uptake ratio of coatings after immersed into the water. From the graph, it shows that sample 2 undergoes pure permeation. The weight of sample 2 increased continuously throughout the whole experiment. It indicates that it has the poorest water resistance as the bonding between the particles is weak to allow the diffusion of water molecules into it. In contrast, samples 1, 3 and 4 showed both migration and permeation. At the beginning of the test, the weight of samples 1 and 4 decreased which deduced that some of the hydrophilic coating materials diffuse out from the coating into the water. After 3 days, permeation processes occurred and causes the weight of this three samples started to increase. This shows that after immersing in water for a period of time, the water can damage the bonding between the flame retardant materials and causes the weakening of the bonding. Therefore, water resistance of coatings would decrease drastically and resulted in the increment in weight.

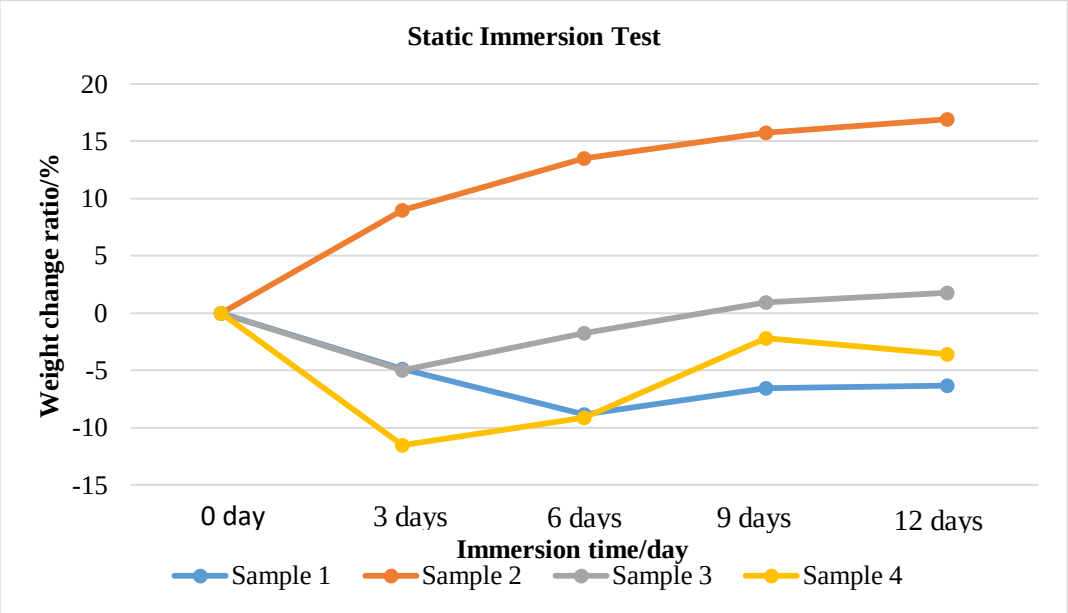

Fig. 5. Relationship between weight change ratio and immersion time

3.4 Adhesion Strength Test

Adhesion strength test was carried out using an Instron micro tester. Table 3.0 shows the bonding strength of coating samples 1, 2, 3 and 4 were 8.15, 2.93, 2.55 and 8.91 MPa, respectively. The sticking areas of all samples are kept constant so the adhesion strength of the coating was depended entirely by the force required to pull off the cylindrical rod.

Table 3
Adhesion strength of coating samples

Coating Sample	Crack Charge, F(N)	Sticking Area, A (mm2)	Bonding Strength, fb (MPa)
1	640	78.54	8.15
2	230	78.54	2.93
3	200	78.54	2.55
4	700	78.54	8.91

Coating 4 exhibited the best adhesion strength with the highest bonding strength of 8.91 MPa followed by coating samples 1, 4 and 2. The adhesion strength of Coating 4 with higher content of PER had better adhesion strength compared to Coatings 1 and 2 with higher content of APP. The adhesion strength of Coating 4 has significantly improvement due to the PER content that provides an effective intrinsic stress transfer. Besides that, adhesion strength of coating can also be affected by the variety of the attributes of the interface region, including its atomic bonding structure, fracture toughness, thickness and purity.

3.5 Scanning Electron Microscopy (SEM)

After conducting the Bunsen burner test, the surface morphologies of char layer formed is observed under the magnification of 1000, 4000 and 8000 by using SEM. The fire protection performance of intumescent coating are strongly depends on the surface morphologies of the char layer formed. Based on the result obtained from the Bunsen burner test, it proved that a thicker char layer formed does not guarantee a best fire protection performance. It also has to depend on the microstructure of char layer formed. Based on the result obtained in Bunsen Burner test, Coating 1 that had the best fire protection with the lowest equilibrium temperature and second thickest char layer formed. As shown in Figure 6, char layer formed by Coating 1 with higher content of APP has uniform and dense microstructure. A uniform and dense structure provide a better fire protection as the fire would not penetrate the coating easily.

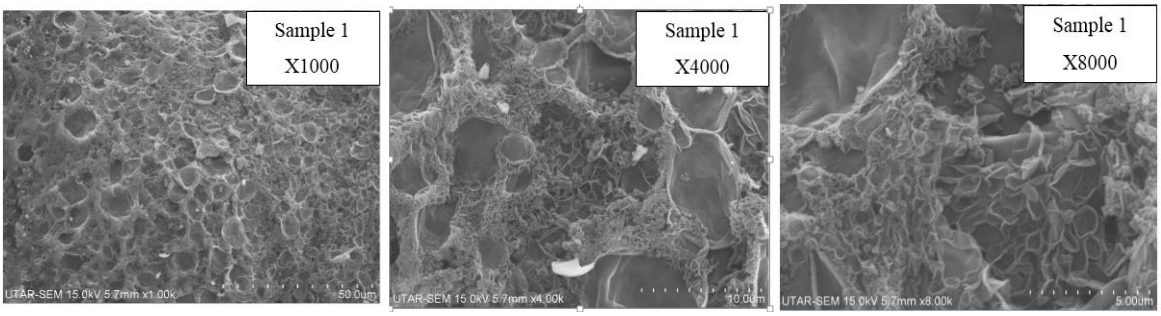

Fig. 6. Surface morphologies of char layer of Coating 1

3.6 Freeze-Thaw Cycle Test

Freeze-thaw cycle test had been carried out to observe the changes in coating layer (freezer > room temperature > drying oven). After placing into the freezer for 1 week, coating samples were left in room temperature for another week before placing into the drying oven for 1 more week. Figures 7 and 8 showed the changes in coatings layer after 3 weeks' time.

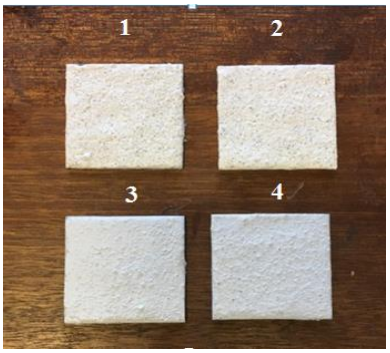

Fig. 7. Sample coatings before freeze-thaw cycle test

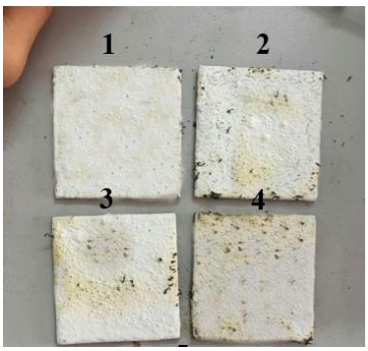

Fig. 8. Sample coatings after freeze-thaw cycle test

It could be observed that some of the coatings have visible changes in colour and surface layer. Among four coatings, the coatings with most colour changes were Coating 4 followed by Coatings 3, 2 and 1. Coating 4 showed colour change and cracks on the surface layer which may result from the expansion of expandable graphite. In contrast, Coating 1 did not show any colour change whereas Coatings 2 and 3 turned a bit brownish after the test. The result proved that Coating 1 had the best weather resistance performance and hence results in better fire protection performances.

3.7 Thermogravimetric Analysis (TGA)

The thermal degradation of four different formulation coatings was determined and analysed using TGA. The coating samples were heated up to 1000˚C to observe the weight lost due to thermal degradation. Figure 9 shows the TGA curves of Coatings 1 (A1), 2 (A2), 3 (A3) and 4 (A4), respectively.

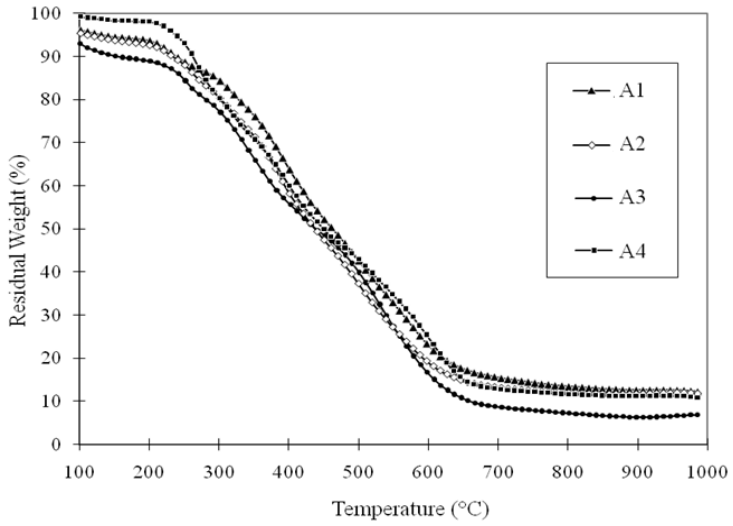

Fig. 9. TGA curves of coating samples 1, 2, 3 and 4

The four coatings showed the similar curve. When the temperature exceeds 215˚C, there was a sharp decrease in residual weight. The decomposition of all samples occurred between temperatures of 215 to 640˚C. The highest residual weight of sample A1 (15 wt.%) indicated that the formulation had the highest thermal stability under air flow in the temperature range of 100-1000˚C compared to samples A2 (13 wt.%), A3 (12 wt.%) and A4 (10 wt.%). The thermal degradation of sample A1 occurs in three main steps: the first step between 215°C and 310°C, corresponding to a residual

weight of 90 wt.%. In the first step, 10 wt.% mass loss was due to dehydration and fusion of PER. The first step of degradation can be assigned to the degradation of PER. The second step occurs between 310°C and 600°C corresponding to a residual weight of 20 wt.%. This step may partly correspond to the polyphosphate decomposing to phosphoric acid, ammonia (NH_3) and water (H_2O) resulting in char formation. There is also the possibility of polyphosphoric acid formation from phosphoric acid with the release of H_2O vapour and NH_3 from the APP. Then, a thermally stable material is formed that degrades from 600°C up to 800°C in the third step and results in a residue of about 15 wt.%. APP forms a phosphocarbon structure that is thermally stable. This stabilized residue acts as a protective thermal barrier during the intumescent fire retardancy process. As a conclusion, an appropriate amount of combination of flame retardant additives could improve the anti-oxidation and thermal stability of the coatings toward the improvement of fire protection performance.

4. Conclusion

In this project, the influence of different ratios of flame-retardant additives on fire protection performance and thermal properties of water-based intumescent coatings were analysed and investigated. Based on the results, Coating 1 consists of 2:1:1 (APP: PER: MEL) showed the best fire protection performance among all the coatings. Coating 1 experienced the lowest equilibrium temperature (157˚C) in Bunsen burner test. This indicated that it has the ability to protect the underlying steel plate against fire for the longest period of time. Besides that, the surface morphology of char layer formed by Coating 1 has a dense and uniform char structure lead to a better fire protection. The results of TGA test showed that Coating 1 (A1) has the best thermal stability due to its highest residual weight (15 wt.%) acts as a thermal barrier. In addition, the adhesion strength of Coating 1 was significantly better than Coatings 2 and 3. Hence, it can be revealed that appropriate composition of flame-retardant additives have proved to be efficient in the protection of steel structures against the fire and weather resistance.

Acknowledgements

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